INTERNATIONAL

Chemical Engineering

and Process Industries

Vol. 32, No. 3 MARCH 1951

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***Subscription rates**: 1 year 4 dollars, 3 years 8 dollars, paid in advance*
England: Leonard Hill Limited, 17 Stratford Place, London, W.1 *U.S.A.: British Publications Inc., 150, East 35th St., New York, 16*

Topics of the Month

Meeting the shortage of metallurgists

THE valuable work of Lord Hankey's technical personnel committee which for some time has been making estimates of the probable future supply of and demand for scientists and technologists, has been taken a stage further with the publication of a report on metallurgists. The report has been made by a sub-committee headed by Sir William Griffiths, chairman of the Mond Nickel Co. Ltd. As was the case with chemical engineers (see INTERNATIONAL CHEMICAL ENGINEERING, December 1950, p. 539), it is predicted that there will be fewer metallurgists than jobs for them over the next five years. In 1949 industry employed 2,600 metallurgists. During 1950-54 some 1,050 will be required to fill posts created by expansion and wastage. A further 300, making 1,350 in all, will be required for education, employment abroad and Government departments. Against this, only 1,150 metallurgists are expected to be available.

This deficit may not seem very serious, but to get a proper view of the situation the quality of the new metallurgists and their suitability for industrial posts must also be considered. In this connection, several firms informed the sub-committee that the majority of graduates, when initially engaged, were lacking in workshop and engineering background, and that additional training, sometimes for two years, was necessary before the new employee became really useful. If this is true, the output of the universities in any one year is only helpful in meeting the industrial demand a year or two later.

There seems to be no burking the fact that graduates must acquire most of their practical production knowledge after leaving college and at industry's expense. This proposition brings the sub-committee to their most important recommendation: That, instead of staying in college after graduation to acquire honours degrees, students aiming at industrial careers should be encouraged either to leave college when they graduate or to spend any further time in college in studies more helpful to their future work. In short, the present obsession with honours degrees should be replaced with inducements for graduates to begin immediately to fit themselves for industrial posts. Industry's part in this proposal must be a general readiness to engage graduates without 'honours' who are suited by temperament and personality for an industrial career outside the laboratory; men so engaged would complete their industrial training on the job.

This suggestion, as with most others which implicitly criticise established practices, is bound to meet some opposition. However, it is difficult to deny that it is a practical answer to the short-term problem of adequately manning metallurgical posts in industry. The long-term solution, of course, is to make metallurgy a career sufficiently rewarding, intellectually and financially, to attract all the men, and women, likely to be needed.

Record exports and imports of chemicals

WHEN the chairman of the Association of British Chemical Manufacturers discussed the industry's export prospects last October, he cautiously predicted that the total value might reach some £100,000,000. Trade returns for 1950 issued recently show that these expectations have been more than fulfilled. The British chemical industry's exports in the Board of Trade classification, 'Chemicals, Drugs, Dyes and Colours,' amounted to no less than

£107,432,000, more than £21,000,000 above the previous record figure of 1949. All sections of the industry have contributed to this achievement. Particularly impressive increases were registered with drug and pharmaceuticals (£22,330,000 against £18,370,000), dyestuffs (£9,330,000 against £8,970,000) and copper sulphate (£2,720,000 against £1,640,000).

In spite of these record exports, the British chemical industry is still far from meeting all domestic demands, as is shown by the fact that 1950 was also a year of record chemical imports; £36,210,000 worth, nearly £11,000,000 more than in 1949. Most of these imports consisted of raw materials and intermediates for the chemical industry itself. Some of the biggest, such as glycol ethers and ether esters and carbon blacks, should eventually be made unnecessary as the British petroleum industry expands. Another big class of imports was potassium compounds, and here again the development of the Yorkshire potash deposits holds the prospect of a steady diminution in the need for imports. Biggest single supplier of chemicals was the U.S.A., which sent more than £9,000,000 worth, compared with the figure of £6,000,000, which was the value of our chemical exports to that country.

Chemical prices

THE rising prices of many chemicals, particularly solvents and heavy acids, tend to obscure the fact that, compared with other industrial materials, chemicals have a very fair record of price stability. At the beginning of 1950 the chemicals and oils index of wholesale prices stood lower, compared with 1938, than any of the other industrial materials and manufactures indices except iron and steel. Apart from coal and iron and steel, the rise in prices of chemicals during the year was the smallest among these indices. However, chemical prices have risen more quickly during the past twelve months than in the two preceding years; 12.6% compared with an aggregate of 5.2%. In judging these figures it should be realised that some important products may now be inadequately represented in the index as it now stands.

The biggest individual increases in 1950 were in the prices of fertilisers and motor spirit. The fertilisers represented in the index are nitrate of soda, sulphate of ammonia and superphosphate, and the prices of these rose on the average by 42% on July 1, when the first stage in the policy of withdrawing subsidies in two approximately equal instalments during the agricultural years 1950-51 and 1951-52 came into effect. The price of motor spirit went up by 9d. per gal. on April 19, as a result of the increased tax, and there were further increases totalling 1d. per gal. on June 1 and September 22. Among the other petroleum products in the index, lubricating oil, burning oil and fuel oil rose in price by 38%, 13.2% and 4.1% respectively during 1950. Maximum prices of sulphuric acid were raised on April 1 and again on November 13, the increases totalling 20% for the grade used in the index. The price of white lead paint rose by 23% during the year. The coal tar products represented in the index rose in price during the year by nearly 16% on the average, while the drugs were 3.2% dearer. The 'other chemicals' series, which like those for coal tar products and drugs has a weight of one, rose by 5.7%, mainly because of increases in copper sulphate and lead acetate prices. The price of household soap was reduced by about 5% in April. However, as reported in our news columns, soap and soap products have now been increased in price.

America's mineral demands

THAT the United States continues to consume her own and some of the rest of the world's minerals at a fantastically high rate is shown by the statistics for 1950 recently issued by the Bureau of Mines. Mineral production last year was 10% higher than in 1949 and only 4% less than the all-time peak in 1948. Its value was no less than $11,725,000,000.

We in Britain, struggling with a fuel crisis and anxiously hoping that our coal mines may yet reach an annual output of, say, 215,000,000 tons, may well ruefully contemplate the 506,000,000 tons of soft coal and 44,400,000 tons of anthracite turned out by the U.S. last year. And this was by no means a record; in fact, as regards soft coal, it was 20% below the peak of 1947. Output of natural gas and natural-gas liquids set new records in 1950 and the petroleum pumped was only 2% less than the 2,000,000,000-barrel record of 1948.

Coming to chemical raw materials, we find that sulphur output reached a new record of 5,200,000 tons, but in spite of this a serious shortage developed in the latter part of the year, a shortage which we and many other countries are now feeling acutely. Production and imports of fluorspar were higher than in 1949 and consumption was at a record level. Principally owing to strikes in the soda ash industry, there was a small decline in total production of salt—from 15,600,000 tons in 1949 to 15,100,000 in 1950. The demand for other chemicals such as barytes, lithium minerals, bromine, borax and lime resulted in an upward production trend. Significant gains were recorded for both phosphate rock and potash compared with 1949. Phosphate rock increased from 9,000,000 tons to a new record of 9,800,000. Output of potash salts, in terms of K_2O, increased from 1,100,000 tons to about 1,200,000, a new high level. Extensions in production facilities were undertaken by established potash companies and at least two new concerns were preparing to enter the field.

America's production of all metallic minerals, except mercury, increased last year. The largest percentage gain over 1949 was reported for tungsten (60%); 20 to 25% each more copper, gold, molybdenum, silver and titanium concentrates were produced in 1950 than in 1949 and 16% more iron ore. Output of lead and zinc increased by 4%.

However, in spite of this huge domestic output of minerals, the U.S. has some serious deficiencies. For instance, although asbestos output increased, it is small compared with requirements. Monazite became increasingly difficult to obtain, owing to export restrictions imposed by foreign suppliers, and new sources of rare earths such as bastnasite are being developed. Domestic production of manganese was less than 10% of the total supply of 2,000,000 tons, while chromite production was negligible, imports reaching 1,500,000 tons. Imports of these minerals from Russia, a major supplier, have almost ceased. In spite of a 63% increase in tungsten output, a shortage of tungsten concentrates arose, caused by increased demand and curtailment of imports from Asia. Cobalt output continued at the same rate as in 1949, but demand was much higher, necessitating substantially increased imports. Almost the same situation prevailed with lead; production increased by 4% to 427,000 tons, but imports rose 22% to 488,000 tons. Similarly, zinc output increased by 4% to 615,000 tons, while imports rose 16% to 428,000 tons.

For the rest of the world the most important of the above figures are those relating to increasing U.S. imports. With

rearmament getting into full stride America's demands upon external supplies will rise still higher and, if things are left as they are, the fact that the dollar is a hard currency means that these demands will be satisfied—at the expense of other countries. If the rest of the world is not to continue to go short it is imperative for us and other nations to devise with the U.S. a system of fair shares of scarce minerals.

The sulphuric acid crisis

THE shortage of sulphur and the consequent introduction of sulphuric acid rationing has resulted in some Parliamentary attention to these vital materials. The Parliamentary Secretary to the Board of Trade told a questioner that every effort is being made to induce the Americans to increase their allocations of sulphur to this country. At the same time the Government is co-operating with industry in projects to convert plants to use materials other than sulphur and to build new plants which do not depend upon sulphur. The possibilities of improving sulphur recovery processes are also being studied.

Another Commons' questioner asked about exports of sulphuric acid from this country and elicited the information that 4,659 tons were exported during 1950, a tonnage which has remained almost constant for the past three years. Malaya and the Irish Republic took the largest quantities.

At this time the statistics of sulphuric acid issued by the National Sulphuric Acid Association acquire particular interest. The figures for 1950 issued last month show that during the year British plants operated at 91% of capacity, producing 1,802,000 tons of acid. Consumption during the year was 1,816,000 tons with, of course, the fertiliser industry taking the lion's share. Stocks of acid at the end of the year amounted to 69,256 tons, compared with 65,417 tons at the beginning. There was a slight increase in all stocks of raw materials in December compared with January, with the exception of sulphur and anhydrite. The following quantities of raw materials were used during the year (tons): sulphur, 357,675; pyrites, 204,987; spent oxide, 194,447; anhydrite, 184,807; and zinc concentrates, 176,365.

New chemical companies

LAST year 380 new chemical enterprises were launched in Great Britain, their total capital amounting to £2,065,940. Two of these were public companies, whose combined capital was £300,000. Of the companies registered during the year whose nominal capital exceeded £100,000, three were chemical companies; one started with a capital of £200,000, the other two with £300,000 each. One of the latter was British Potash Development Ltd., an enterprise evidently connected with the recent discovery of potash deposits in Yorkshire.

These facts about new British chemical companies come from statistics compiled by Jordan & Sons Ltd., the London company registration agents. These show that company registrations continue at a level far higher than before the war. However, 1950's crop was smaller than 1949's—13,278 companies capitalised at £67,762,072, against 13,755 capitalised at £76,496,452. By far the greater number (13,095) were private limited companies.

The biggest class of new registrations was that concerned with food, 1,354 companies with a total capital of £7,186,855. Other new companies in the process industries were: oil, 24 (£276,800); plastics, 55 (£187,900); rubber, 33 (£198,600); textiles, 650 (£4,675,800); bricks, cement, etc., 86 (£574,550); and mines and quarries, 42 (£1,790,000).

More money for standardisation

FIFTY years ago the Institution of Civil Engineers appointed a committee of six members for the standardisation of steel sections such as girders. It was estimated that their success in this reduced costs by 5s. a ton and saved industry £1,000,000 a year. From this has developed the British Standards Institution of today, working through some 2,000 different technical committees, and responsible for about 1,500 general standards, over 200 special standards and some 30 codes of building practice. Even since 1939 the number of general standards has almost doubled.

Current annual expenditure is roughly £220,000—less than one-quarter of the estimated saving from the work of that first 1901 committee on steel sections.

It is estimated, however, that with further advances in standardisation this expenditure will double over the next few years and it was in view of this that, in June 1949, the President of the Board of Trade appointed a committee under the chairmanship of the Hon. Geoffrey Cunliffe to consider the Institution's organisation and constitution.

This committee has now issued its report ('Report of the Committee on the Organisation and Constitution of the British Standards Institution,' H.M.S.O., 1s.).

The report refers to the recommendations of the 1949 committee under Sir Ernest Lemon on standardisation in the engineering industry; to the report of the group which went in 1949 from the Anglo-American Council on Productivity to study 'the policy of simplification' in the U.S.A.; to the setting up by the O.E.E.C. of an 'advisory group for increasing productivity through standardisation'; and to moves in several industries for certification-marking schemes dependent on suitable standards. There is no doubt, they report, that industry increasingly recognises the economic importance of standardisation, that it will proceed by consent, and that the B.S.I. will need to be expanded to meet the increase in the volume of work which this growing interest will entail. 'We regard it as self-evident that an expansion of the B.S.I. is urgently necessary.'

On the question of finance, the committee praises the free services given by industry and Government, which 'vastly exceed their actual cash contribution.' In one instance where a piece of research work was accurately costed the result was in the region of £1,000.

On subscriptions from private individuals the committee offer no comment. From other subscribers they recommend 5 guineas as a lower limit. For commercial undertakings the committee recommend a scale of subscription based on the number of employees, though this would be intended only as a guide to the sum that should be paid by a firm 'wishing to contribute its share.' While many firms make substantial contributions to the B.S.I., the report points out that only some 3,000 to 4,000 manufacturing firms subscribe to the B.S.I.

The Institution has received a contribution from the Government every year (except 1909) since 1904. The contribution at present stands at £90,000. A formula arrived at in 1943 for relating Government to industrial contributions is already out of date, since it would produce considerably less than is needed. For the year 1951-52 they recommend a minimum Government contribution of £90,000, and thereafter a contribution equal to the income from industrial subscriptions, subject to a maximum of £150,000. Such a contribution, they say, is justified on two grounds: Firstly, the Government is a large purchaser

of goods of all kinds; secondly, it represents the public as consumers of goods which, in the absence of standards, might be dearer or less serviceable.

At the same time, too large a subscription would prejudice the Institution's autonomy, and the Committee thought it unwise to suggest a Government contribution in excess of that subscribed by industry.

The committee finds that the constitution and organisation of the B.S.I. is well adapted for carrying out its present work and the increased work which may be expected to fall upon it. The B.S.I. should remain under the control of its own General Council and retain its present status as a body incorporated by Royal Charter.

The B.S.I. has expressed general acceptance of the recommendations of the committee, and the Government is now considering action on recommendations which raise Government issues.

British electronic sterilisation project

SINCE we published the note last month on American work on the electronic sterilisation of food (p. 61) we have learned of the formation of a British company which proposes to investigate these claims and, if they prove well-founded, to develop suitable processes in association with American workers. The company is called Irradiation Research & Development Ltd., and among its directors is Sir Robert Watson-Watt, F.R.S., the distinguished electronics engineer.

Dr. C. P. Stewart, managing director of the firm, tells us that in his view, of all possible forms of sterilisation and preservation by irradiation, high-speed electrons afford, at present, the most hopeful line of approach, being free from the limitation of low penetration as in the case of ultra-violet irradiation, and being both efficient and freer from danger to operators than *x*-rays. The use of high-speed electrons demands high-voltage generators giving voltages of the order of five to ten million volts, of which three types are available. It also requires special 'cathode ray' tubes which can and have been produced. In America, at least two teams are working on these lines and one has gone so far as to construct a machine which successfully sterilises such materials as pharmaceuticals, which are fed to it, ready packed for the market, on a conveyor belt.

Irradiation processes will not completely replace existing methods of preservation, because their inherent qualities impose limitations. For instance, they can be applied only to material which can be irradiated in relatively thin layers, or which needs only surface treatment, and to material which can be treated in reasonably large quantities. However, bearing in mind that the older and still generally used methods of preserving and/or sterilising food frequently fail to satisfy the condition of avoiding objectionable changes in the appearance and taste of food, it seems that electronic sterilisation fully justifies intensive investigation and development.

Japan's cement industry

OUTPUT of Japan's cement industry, though still below the pre-war peak level of 5,700,000 tons p.a., will exceed the target figure of 4,100,000 tons for the fiscal year ending March 1951 by some 100,000 tons, it is anticipated. In the next fiscal year, output is expected to expand by 10%.

Before the war, Japan was the world's fourth largest cement producer. By 1946, the year following her surrender, output had fallen to 927,000 tons. Since then, annual output has steadily recovered—to 1,232,000 tons in 1947, 1,843,000 in 1948 and 3,271,000 in 1949.

Exports, which before the war constituted 10% of production, ceased completely in the early post-war years. In 1948, however, 140,000 tons were shipped abroad, and in the following year 485,000 tons. Exports in 1950 were expected to approximate 600,000 tons, which would have compared very favourably with the pre-war years, but the suspension of shipments to Korea caused some decline.

The distribution of exports corresponds closely to that of the pre-war period. The Philippines, India, Indonesia, Thailand and (until recently) Korea, are still among the principal markets. Shipments are also made to Africa and South America. The prevailing export price of Japanese cement is around $13 (U.S.) per ton f.o.b., which is a little higher than the average domestic price for the ordinary Portland grades.

With large deposits of good-quality limestone and adequate supplies of coal, Japan is almost wholly self-sufficient in the raw materials needed for cement manufacture. The price of coal, however, has now risen to a level which the industry considers disadvantageous compared with the fuel costs of cement producers overseas.

The industry is now concentrated into 15 companies operating 33 factories with a theoretical capacity of 6,000,000 tons p.a. The companies are all private corporations, independent of the Government or foreign interests both in capital and management. The majority of companies are solely cement producers; in a very few cement is a subsidiary manufacture.

The Widnes chemical tradition

THE story of the past 100 years of the Lancashire town of Widnes forms a substantial part of the history of the British chemical industry. It was in 1847 that John Hutchinson built his Leblanc soda factory in the then village of Widnes; in the same year and in the same community John McClellan of Liverpool established his North British Chemical Co. and manufactured borax and, later, alkali.

These men were the forerunners of a line of chemical giants—Muspratt, Robinson, Brunner, Mond and Hurter—who between them established the heavy chemical industry and made Widnes a great name in the chemical chapter of the industrial revolution.

Last year Imperial Chemical Industries Ltd., heirs and guardians of the Widnes chemical tradition, organised the celebration of the centenary of chemical manufacture in Widnes. At an early stage in the preparations for the celebrations it was decided that an enduring record should be made of this story. This task was given to Dr. D. W. F. Hardie of the research staff of I.C.I.'s General Chemicals Division. Last month Dr. Hardie's book was published by I.C.I. with the title 'A History of the Chemical Industry in Widnes' (pp. 250, including index, 21s.).

Drawing primarily upon the archives of the Widnes companies now merged into I.C.I. and upon conversations with descendants of the chemical pioneers, the author has written a fascinating story of people, processes and plants. It is far from being merely a technological history; it is a drama for which the plot is provided by the famous soda process invented by the ill-starred Nicholas Leblanc. The growth and decline of his process is largely the story of the manufacturers, chemists and inventors in the factories between the canal and the marsh of Widnes.

Design and Manufacture of Pressure Vessels

Manufacturers' and Users' Views on British Standard 1500/1949

Although complete community of interest now seems to exist between users, inspecting authorities and manufacturers of pressure vessels, the manufacturer is mainly interested in efficient design and fabrication, whereas the user asks that his pressure vessel be provided as cheaply as possible but yet operate without danger to personnel or plant. These different viewpoints were discussed in two papers dealing with fusion-welded pressure vessels in reference to British Standard Code 1500 read at the third conference on chemical works safety sponsored by the Association of British Chemical Manufacturers and organised by the Industrial Division of the Royal Society for the Prevention of Accidents at Scarborough recently. Below are summaries of the different points of view put forward by a manufacturer and a user.* Other points raised when this Code was discussed by the Institution of Chemical Engineers were reported in INTERNATIONAL CHEMICAL ENGINEERING, November 1950, pp. 519-521.

Manufacturers' viewpoint

THE application of British Standard 1500/1949 and its value to the manufacturer were discussed in a paper by W. R. Chipperfield, Babcock & Wilcox Ltd., and read by his colleague, A. Webb. The progress of pressure vessel design was considered first.

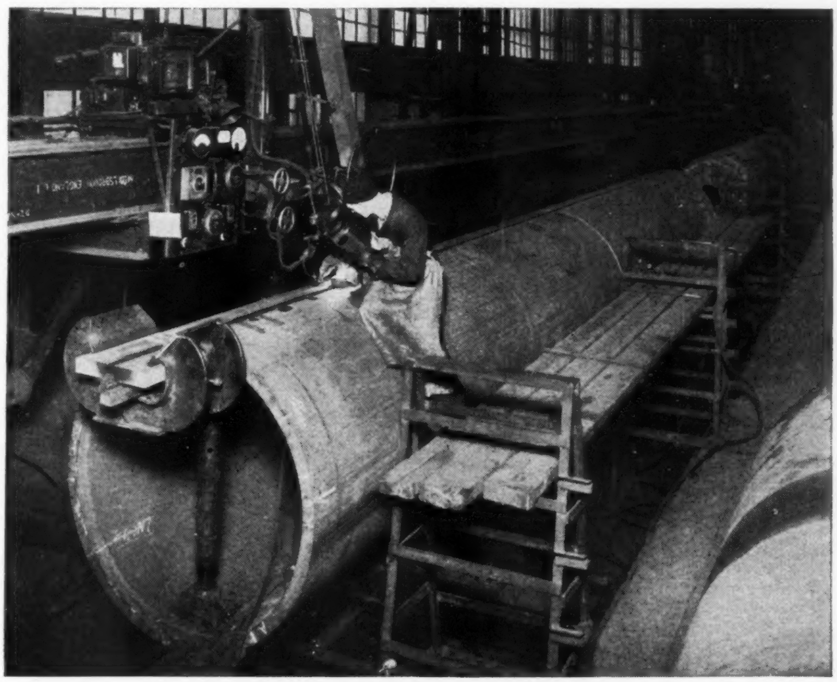
In early days pressure vessels were almost exclusively manufactured by riveting, then solid forged drums were considered. For large applications and in search of economy, several welding methods were employed. The development of an automatic welding machine led to research in metallic arc fusion welding, which is now the most generally adopted technique. With modern methods and testing procedure, such as are laid down in B.S. 1500, very satisfactory vessels can be produced, and for many applications welded drums of this type have superseded riveted vessels. The issue of this provisional code, therefore, has catered for the majority of pressure vessels which will be required. Steels are, moreover, the only materials with sufficiently high tensile strength at reasonable cost which can be used in quantity and the author explained the procedure adopted in a modern plant to manufacture such vessels.

After giving technical details concerning the testing of the metal sheet, the methods of carrying out the weld and the testing of the joint, the author stressed the need for skilled and efficient welding operatives. Considerable attention must be paid to the training of welders and, in devising the training schemes, careful attention must be given to procedure, which must be broad enough to cover all the ground but yet simple enough to be readily assimilated. Periodic check testing of the welders' ability was also essential. Check on the setting up, correct storage and issue of electrodes and the adequate maintenance of welding machines were also desirable factors. Attention to these matters gives very good results, and to illustrate the reliability of welding methods it is interesting to note that, in the plant considered by the author, where such attention is paid, the total of repairs found necessary in the main seams of welded vessels has been equivalent to about only 10 in./mile of welding.

With the demand for various types of vessels, research, sometimes of a fundamental nature, has to be undertaken. Several recent studies have been made of the stress distribution in pressure vessels and electric strain gauges have proved useful. Experiments are also proceeding to determine the best form of mandoor opening in dished ends. Other work concerns the suitability of various alloy steels for use in gases at high temperatures.

The Code

The regulations necessary to standardise the design and manufacture of these vessels are very completely explained in B.S.



Multi-layer welding of longitudinal seam.

*Extracted from the 'Proceedings of the Third Conference on Chemical Works Safety,' published by the Association of British Chemical Manufacturers and obtainable from them, price 5s., post free.

1500. The Chemical Engineering Industry Standards Committee of the British Standards Institution, containing as it did, representatives of the users, manufacturers and inspecting authorities, was able to consider the interests of all these parties and the complementary views expressed enabled a mutually satisfactory Code to be produced. A vast amount of data had to be sifted and correlated before agreement could be reached by the members of the committee on the various clauses. In some cases the requisite data was not available and original research had to be carried out.

The Code differs somewhat in make-up from other British Standard Specifications because of the nature of the subject. After a general explanation and notes on the use of the Code, Section 2 covering materials and stresses follows. This part was considered of sufficient importance for a separate specialist panel to be set up to consider the matter. After examination of much information which was made freely available by the steel-makers and others, it was decided generally for ferrous materials to base design stresses on a constant factor of safety of 4 up to a temperature of 650°F. and on creep data for higher temperatures. The importance of creep decreases with temperatures below about 800°F., so that, between this figure and 650°F., the values of stress are gradually raised from the value at 800°F. to that at 650°F. The tables of design stresses for non-ferrous materials are still being considered and the figures will be included in later editions.

Section 3, dealing with design, has been deliberately prepared using a discursive style appropriate to a Code. The terms 'design,' 'temperature' and 'design pressure' have been carefully defined and, in connection with the last, it will be noted that a recommendation is given that there should be a margin between the design pressure and the normal operating pressure to prevent frequent blowing off of the safety devices.

After dealing with corrosion, erosion and protection, regulations governing scantlings are set out. Three classes of welded vessels are included and are known as:

(1) Severe-duty vessels.
(3) Medium-duty vessels.
(3) Light-duty vessels.

Regulations governing plate percentages are similar to those in B.S. 1113.

With regard to the actual calculation of shell thickness, the 'mean diameter' formula is used for vessels having a shell thickness not greater than 10% of the internal diameter, and for greater thicknesses the Lamé formula is used. The most reliable method of computing the pressure at which a cylinder ceases to behave elastically has not yet been resolved. The simple theory assumes that it occurs when a maximum principal stress is reached, and this is probably true for brittle materials, but it is possible that a ductile material such as mild steel follows the maximum-strain-energy relation more closely. Nevertheless, the maximum stress theory calculated by the Lamé formula has been widely used, so that there is ample experience of behaviour of vessels designed in this manner and for the proportions of vessels to which this Code applies, the differences between the theories are not great, having in mind the factors of safety involved; it is therefore felt that the inclusion of the Lamé formula is justifiable.

With regard to the design of ends, it will be noted that thicknesses for given conditions depend upon the actual shape of the end. It has been amply demonstrated that this is a rational procedure and much work has been done on this subject. The rules themselves are framed on certain recommendations which followed the work of Siebel and Korber at the Kaiser Wilhelm Institute for Steel Research in 1925, confirmed by later investigations in this country.

After proceeding through the regulations concerning other shapes of ends and the designs of tube nest construction and of openings and branches, a series of typical welded connections is given. In preparing these typical connections a further panel was set up and the most satisfactory types from the different practices have been included.

With regard to flanges, following the general practice throughout the Code, reference has been made to B.S. 10. Other regulations dealing with the design of jacketed vessels complete this Section 3.

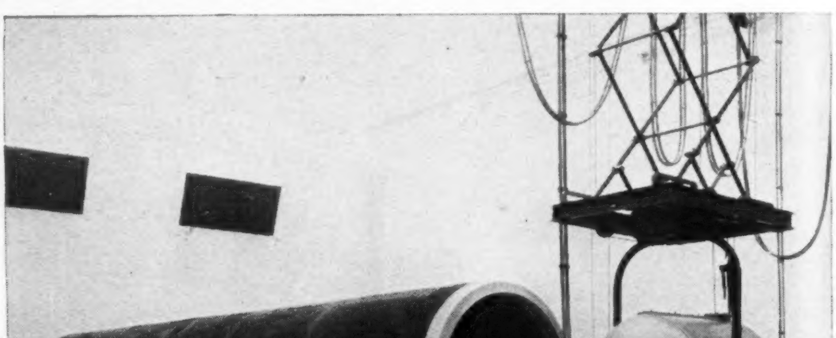
Section 4, 'Manufacture and Workmanship,' closely follows corresponding clauses in other related specifications, particularly the rules relating to the design of fusion-welded boiler drums in B.S. 1113.

The same correspondence with other specifications occurs in Section 5, 'Inspection and Tests.'

Protective devices

Section 6 deals with 'Protective Devices.' All pressure vessels must be protected, except those connected with an adequate system of piping having no shut-off valves to other vessels having safety valves, or vessels whose pressure originates solely from a positively controlled source. This protection may be by safety valves, or by a combination of safety valves with rupture discs, or in special cases by rupture discs only. The safety valve clauses follow, where applicable, the relevant clauses in B.S. 759, 'Valves, Gauges and Fittings for Land Boilers.' In determining the capacity of safety valves the steam duty is used and the required capacity multiplied by a capacity factor which is obtained from Figs. 62 and 63. The total capacity of the safety device must be sufficient to discharge the maximum quantity that may be generated or supplied to the vessels without the pressure rising more than 10% above the permissible working pressure. In setting the safety valves, one must be set to operate at the maximum allowable working pressure and any other valves set at not more than 5% above this pressure. With the vast experience with steam boiler safety valves which stands behind the provisions of B.S. 759, it is considered that these Code requirements will prove quite satisfactory.

The use of rupture discs is recommended only where either pressure fluctuations of



2,000,000-volt *x*-ray equipment.

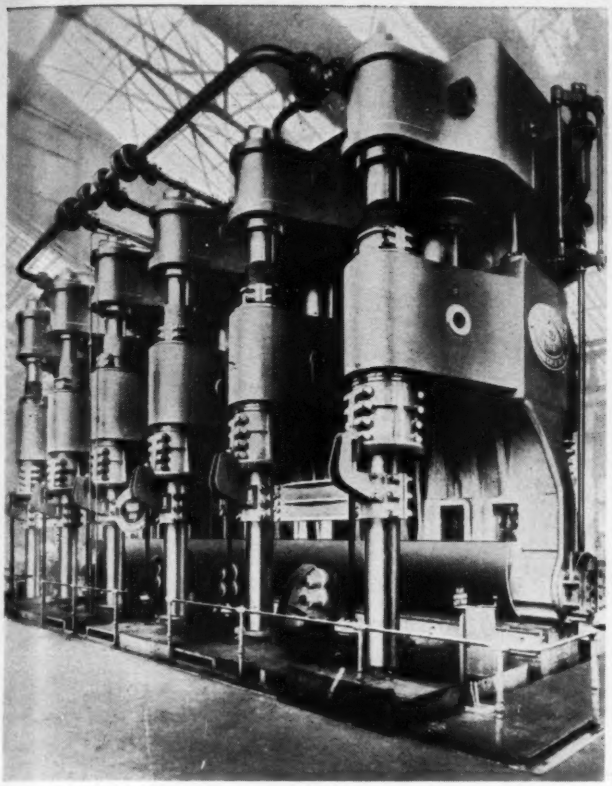
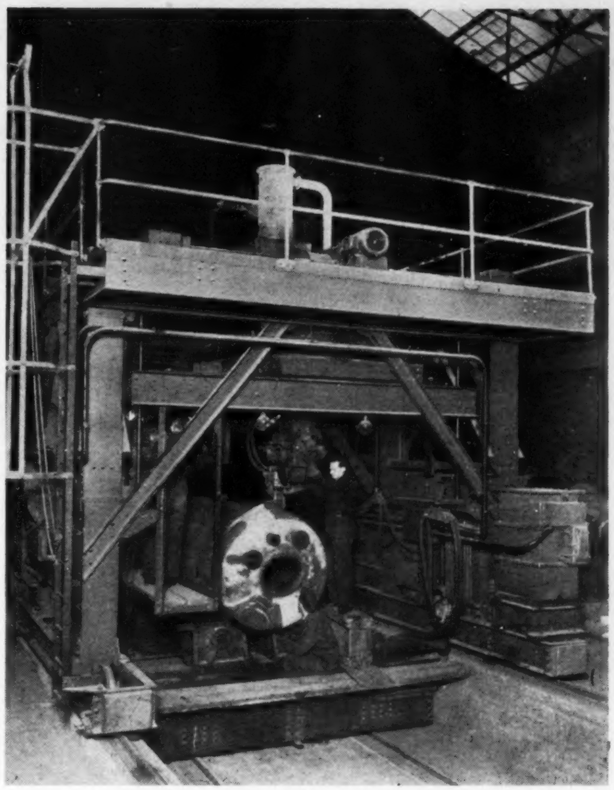


Plate-bending press.



Unionmelt machine used for submerged-arc welding.

These and photos on pages 111 and 112 by courtesy of Babcock and Wilcox Ltd.

an explosive nature are to be dealt with or where the working fluid has properties which make the use of a safety valve impracticable. Where fitted, rupture discs must be replaced frequently to avoid premature failure and, if no safety valves are fitted, they should be designed to burst at a pressure not exceeding the safe working pressure. In cases where rupture discs are fitted as well as safety valves to deal with exceptional conditions, the valves must be designed for the full duty and the disc designed to rupture at a pressure at atmospheric temperature not exceeding the hydraulic test pressure. Regulations are included in the Code dealing with the correct installation of valves and an important provision concerns the use of stop valves beneath the safety valves. These are only permitted if a spare safety valve is provided and only one handle must be supplied for all the stop valves, and this handle must be so designed that it cannot be removed until the valve is opened. Provisions are also set out regarding the design of escape piping.

The Code concludes with a section dealing with service inspection and repairs and an appendix dealing with mechanical tests carried out on the test plates representing welded seams.

Advantage of having a Code

A Code of practice issued by an authority such as the British Standards Institution covering certain products is of great value to manufacturers. Besides the obvious advantages which accrue from the ability to standardise certain details and works procedure, there are two other advantages which are not so apparent. In chemical engineering the ideal plant which industrial chemists would like has frequently to be modified so that its manufacture is practicable and economically justifiable. In vessel design the existence of a Code covering details enables practicable designs to be built up and helps both the client and his contractor. Again, in competition, especially in the export market, it is of great assistance if a manufacturer can quote to a British Standard. Hitherto, quotations which have been submitted have had to be accompanied by detailed specifications which made it difficult for some clients to assess their worth, or, alternatively, manufacturers have quoted to other Codes such as the API.ASME Code and explained modifications which had to be made to suit British manufacture. In either case clients are liable to be less impressed than they might have been by a straightforward declaration of conformity with a British Standard.

It will now be appreciated why the manufacturers welcome this Code and were very willing to assist in its preparation. The difficulties which had to be surmounted were considerable and the committee have issued it in a tentative form for a year's trial. It remains for the industry to use the Code and report on their experience so that a final document can be issued which will be in every way a credit to British engineering. Such an achievement will enhance our reputation and go a long way towards the point where chemical manufacturers can obtain equipment designed and built to a first-rate British Standard Code.

Users' viewpoint

British Standard 1500 can be considered to have as guiding principle safety with economy. J. W. Strawson, Shell Refining & Marketing Co. Ltd., took this principle as his theme, high-lighting aspects of the Code which are of particular significance from the point of view of the user or of special interest because they represent a departure from the usual pattern of British Standard Specifications relating to pressure vessels.

Although tentative efforts to produce a British Standard Specification for fusion-welded pressure vessels were made as early as 1939, a really serious start towards the preparation of what is now B.S. 1500 was not made until 1940, when the Council of the Institution of Chemical Engineers wrote to the British Standards Institution stressing the fact that the scantlings laid down or being proposed for the various

British Standard Specifications dealing with welded pressure vessels were very much more conservative than those jointly issued by the American Petroleum Institute and the American Society of Mechanical Engineers. This, it was claimed, was having an unfavourable effect on export trade, since vessels built to British specifications could not compete in price with those made to American standards.

The American Society of Mechanical Engineers' Boiler Code was carefully examined and the obvious thought that the ASME Code might be adopted as a basis for a British Pressure Vessel Code received serious consideration. Although it might be said that B.S. 1500 has followed the American trend of thought regarding permissible design stresses for working temperatures of the order of 600°F. and higher, it can be fairly claimed that in recent years America has drawn in line with British practice over a much wider field of industrial applications included in the temperature range up to 450°F. or so, say 250 lb./sq.in. saturated steam conditions.

[Photo: Shell Refining and Marketing Co. Ltd.

Two 35-ft diam. spherical vessels during construction.

Range of vessels covered by Code

B.S. 1500 is intended to be applicable to the design and construction of all vessels except those where the pressure so nearly approximates atmospheric that they are by common consent described as tanks. It covers a wide range of sizes and operating conditions.

Design stresses

In criticising the permissible design stresses the author notes that a general hardening of opinion is discernible in favour of a stress value around 4.5 to 5 tons/sq.in. for a butt-welded joint whose qualities have been proved by a simple set of tensile bend and 'nicked break' tests.

The term 'joint efficiency' is a misnomer, because it is impossible to calculate a straightforward 'efficiency' factor for a welded joint in the same way as is usual for riveted connections. Joint efficiency factors might better be described as weld quality factors.

Site-welded vessels

The construction of large vessels under site conditions is something new to this country; the total number of such vessels in Great Britain in operation at the moment, mainly in the gas industry, is believed to be less than a score, but this number will appreciably increase when plants now being built as part of the oil refinery expansion programme are brought into operation. Although the pressure conditions are necessarily low, the stress conditions are severe, because of the large diameters involved, and a high standard of weld quality is required. In the writer's opinion all major site welds in pressure vessels should be subjected to some form of non-destructive examination. Radiography is still the most common method, but other techniques such as ultrasonic echo methods, which have been used for similar applications in structural engineering, are showing promise and may prove more convenient for field use.

Plate preparation for welding

When the drawing of any welded vessel is examined, the most striking feature is usually the number of details of typical welds drawn out to a large scale. The treatment which the Code affords this aspect may appear to some to be irrational, the preparation required for the main seams being scarcely touched upon, while the requirements for weld preparation for branch pipes and similar attachments are covered in precise detail. This does not arise from oversight or neglect, but is the result of deliberate policy. It is easy at times to pay so much attention to the details of weld preparation that the fundamental requirement, a sound, welded joint, is lost sight of.

The actual shape of the plate edge preparation is of little significance, provided that the finished *weld* is sound; probably, of course, the smaller the amount of added weld metal required the better, as residual stresses may then be expected to be reduced. If, therefore, the quality of the finished welds can be checked, it is best to leave the manufacturer free to adopt whatever form of preparation he has found best suited to his particular manufacturing technique. This condition does, in fact, apply to the main seam welds, but the welds attaching branch pipes to a vessel shell are notoriously difficult to check; even radiography does not provide a very satisfactory examination and is not, in practice, considered worth while. The preparation for branch pipe attachments should, therefore, be such as to make the production of sound welds as easy and near foolproof as possible. Given reasonable skill on the part of the welding operator, the preparation illustrated on pages 94, 97-101, 118 and 119 of B.S. 1500 satisfy this requirement.

Bolts

The most important bolts connected with pressure vessels are those in bolted flanged joints. The writer believes that the rules given in the present version of the Code, and all similar publications, for dealing with these details are of a very hit-and-miss character, having been handed down through the years. However, it must be admitted that, in practice, they usually result in a 'hit,' by virtue of the low stresses laid down and the fact that full-faced joint flanges (which are the only ones covered in details as yet by the Code) normally have a soft jointing material. Even if a 'miss' is discovered it can often be turned into a 'hit' by use of some jointing paste and a length of pipe on the spanner. Rational design calls for something a little more scientific than this, and the writer suggests that no procedure for the design of bolts can be defended which does not give some consideration to the force which has to be exerted on the gasket in order to seal the joint.

Certification of vessels

It is the regular practice for pressure vessels to be constructed under the general supervision of an independent inspecting authority employed by the purchaser or under the supervision of the purchasing company's own engineering staff, and this procedure does apparently succeed very well in keeping the manufacturers up to scratch. However, the users' main guarantee of safe vessels lies in the integrity of the manufacturers, the best of whom have a genuine appreciation of their responsibilities and operate in accordance with the spirit of the various specifications and the Codes they are asked to work to in producing vessels. It is fortunate that this is so, because there is no doubt that an unscrupulous manufacturer *could* produce a bad vessel which apparently complied with the strict letter of any specification. Anticipating the day when the industrial conscience might not remain so effective under competitive trading conditions, the writer feels that the British Standards Marking Scheme should be applied to pressure vessels, in addition to the range of products already controlled by the scheme.

Capacity of safety valves

In the event of an emergency the safety of a vessel may be wholly dependent upon the satisfactory operation of the pressure-relieving valves, and it is important that the valves fitted should be capable of passing the maximum amount of gas, vapour or liquid necessary to limit the pressure to a safe level under the worst combination of conditions which may occur in practice. It is, of course, not feasible to carry out rating tests on all types and sizes of valves using all fluids of industrial interest, and recourse must necessarily be had to procedures which will permit the capacity of a valve dealing with any fluid, to be estimated from its performance when dealing with saturated steam. In the Code, such procedures are adopted.

Discussion

In the discussion which followed the reading of the two papers some interesting views were expressed.

Mr. J. Eyers (Vulcan Boiler & General Insurance Co. Ltd.) stated that certification authorities took a lively interest in the proposed new chemical pressure vessel Code and he felt that one of the effects of the Code was that pressure vessel design would be concentrated in the hands of specialist firms, which was very good.

Mr. Strawson would like to have seen B.S. 1500 confine permission to describe vessels as being made to B.S. 1500 to manufacturers who were under the control of the B.S.I. certification scheme. Mr. Eyers pointed out that if supervision were introduced over manufacturers, there would be trouble if the right to use the Code stamp were taken away at any time and thus put them out of business.

Prof. D. M. Newitt (Imperial College) stated that, whatever might be thought of the Code, it did have value on focusing attention on the weak points in design. A point which was not, in his opinion, adequately dealt with was the question of stress due to temperature gradients. In vessels using thick plate it was possible to get transient temperature gradients of several hundred degrees, and it could be shown that the kind of stresses set up were those which might extend the metal beyond its elastic yield strain. In design one had to pay particular attention to ensure that local gradients could not be set up at points of entry into the vessel.

Mr. J. D. Usher (A.P.V. Co. Ltd.) said that his concern had been basing designs on that Code during the past year, and a number of cases had been brought to light where the provisions of the Code conflicted with their standard design practice. His impression of the Code was that it had been compiled chiefly to apply to the heavy type of mild-steel vessel and had not paid sufficient attention to the lighter type of vessel in stainless steel and non-ferrous metals.

Mr. E. G. Ryan (I.C.I. Ltd.) said that a safety aspect which should have been covered in the Code was that relating to the size of manholes in rubber-lined vessels. He said it was common to find rubber-lined pressure vessels being made with manholes smaller than 18 × 16 in.; thus, when it is necessary to repair the rubber lining (an event which should be allowed for, no matter how unlikely), the repairer who has to use solvents classed as toxic substances had either to obtain an exemption from Clause 27 of the Factory Act or fail to meet the requirements of the clause. It was obviously undesirable to take advantage of an exception if the hazard can be reduced, even at some extra expense, by the provision of a removable end (by virtue of the small diameter of vessel involved); thus, where it is not practicable to fit 18 × 16 in. openings, it would be desirable to provide a removable end.

Low-pressure Polyester Resins

THE characteristics of the low-pressure polyester resins were recently outlined by Dr. J. D. Robinson, American Cyanamid Co., speaking at a New York meeting of the Society of Plastic Engineers. During the war, Dr. Robinson related, these resins were taken directly from the laboratory and put to use in aircraft parts. In the autumn of 1946, when war contracts were being terminated, there were very few civilian uses known for polyester resins, with the exception of paper-base decorative laminates.

These new resins are the only thermosetting materials which have a range of properties extending from a highly rigid, brittle-type resin to flexible materials which are similar in some respects to vinyl compounds. All of the resin reacts without the necessity of removing solvent. They are fluid and easily handled because of the wide range of viscosities available. Special types of resins were available for fire resistance; crystalline wax-like resins were available for use as hot melts.

Polyester resins cure quickly. They are exothermic in their reactions, which means that the power necessary to cure them is reduced. They are quite fluid and capable of flowing rapidly, can be handled and formed with very low pressures.

Two problems involved in the use of low-pressure polyester resins were discussed. They are the fact that there is shrinkage in passing from the liquid, monomeric stage to the rigid, cured condition, and the control of exothermic reaction to prevent the resins reacting so vigorously that the quality of the plastic part is damaged. These problems are being worked out and additional information is being obtained daily.

Inert mineral fillers have been found useful in correcting some of the problems which are met with in the industry. Such things as trays, instrument cases, skylight panels, housings for vaporisers, sterilisers and lamp shades are examples of products now being produced from polyester and glass mat. Paper-base laminates, made continuously on sheets, are used in many places where decorative surfaces of good abrasion resistance is indicated.

Some resins are being used for industrial castings to make such things as electrical capacitators, fishing rods, etc. Electrical instruments are potted in polyester resins. Armatures for electric motors, signal devices for traffic lights, etc., are other examples of the usefulness of these resins in the electrical field. In addition to surfacing paper-based laminates, polyesters are also used for surfacing masonite, plywood and similar structural board.

In discussing methods of fabrication, Dr. Robinson used polyester resin and glass mat as an example. Early work was done by laying the mat, saturated with resin, on a plaster or wood mould and covered with a separator sheet and a rubber blanket. The pressure was applied by evacuating the system and cure was accomplished by heating in an oven.

The next step was the used of matched metal dies or combinations of a metal mould and the application of fluid pressure from rubber bags or other similar sources. More recently, the trend has been to match metal moulding with cut-off rings designed to give positive pressure on the moulded part during the cure operation. In this way, pressure is applied on the liquid resin rather than on the glass mat fibres and parts with good surfaces have been developed.

Explosives Manufacture

By R. T. W. Hall, B.Sc., Ph.D., A.R.I.C.

(Lecturer in Chemical Engineering, Birmingham University)

Although the subject of explosives is nearly always connected with military applications the production of explosives in peace-time has reached the proportions of a major chemical industry. The modern age is indebted in many ways to the rightful application of explosives, from the every-day blasting operations involved in mining and similar industries, to such gigantic undertakings as the construction of the Panama Canal. Furthermore, some of the modern explosives have found other uses of an entirely different nature such as the medical use of nitroglycerine. In this article* the author deals with some historical, chemical and engineering aspects of explosives manufacture, a task on which he was engaged for several years during the last war.

THE oldest explosive known is gunpowder, which was first recorded by Roger Bacon, a Franciscan monk of Ilchester, Somerset, in 1242. Bacon published his discovery in Latin in anagrammatic fashion to keep the information secret.[1] His original mixture consisted of 7 parts saltpetre, 5 parts each of hazel twigs and sulphur.

Gunpowder was first used in military operations by the English forces at the Battle of Crecy in 1346, and remained the only propellant until 1880, when smokeless powders began to come into use. It still has, however, both military and peaceful uses, being easily manufactured. It finds use in bursting charges for shrapnel shell, as a primer for propellants, for fuses and for pyrotechnical and sporting use. Modern compositions approximate to 75% KNO_3, 15% C (charcoal), 10% S.

The chemical changes involved in the decomposition are complex, but approximate to the equation

$$2KNO_3 + S + 3C = K_2S + 3CO_2 + N_2.$$

The addition of starch-wax granules, or certain sulphates, as cooling agents has produced modified gunpowders, and in recent years ammonium nitrate has replaced the potassium salt as the oxygen supplier in many explosives, *e.g.* the mining explosive 'ammonal.'

Another type of modified gunpowder is to be found in the 'liquid oxygen explosives,' where the original nitrate is replaced by liquid oxygen, giving a powerful explosive, used to a large extent in the construction of the Simplon tunnel in 1899.

Types of explosives

At this stage, it must be pointed out that there are essentially three major types of explosives required:

(*a*) *Propellants*, which have a controlled rate of combustion, insufficient to shatter the gun barrel but sufficient to eject the projectile at high speed;

(*b*) *High explosives*, which have maximum burning rates, and generate maximum power;

(*c*) *Detonators*, to initiate the decomposition of other more 'stable' explosives.

The *explosive properties* of a chemical depend on the combustibility of the substance and primarily on two reactions:

$$C + O_2 = CO_2$$
$$H_2 + \tfrac{1}{2}O_2 = H_2O.$$

The main oxygen supply is found in the nitro ($-NO_2$) group.

Explosive power depends on:

(*a*) The amount of energy which can be liberated;

(*b*) The rate of combustion;

hence the carbon atom should be fairly close to the available oxygen atoms.

Nitro groups occur in three main forms:

(*a*) Nitro-hydrocarbons (*e.g.* T.N.T.);

(*b*) Nitro-esters (*e.g.* nitroglycerine);

(*c*) Nitro-amines (*e.g.* R.D.X.).

Propellants

Modern explosives are considered to have originated just over a century ago, with the discovery of nitroglycerine by an Italian, Sobero, who nitrated glycerine, which had previously been discovered by Scheele about 1780. Thus the first modern explosive was developed from raw materials such as olive oil and other vegetable oils. Nitroglycerine is still manufactured by the nitration of glycerol and is strictly a nitrate ester, rather than a true nitro-compound:

$$\begin{array}{llll} H_2.C.OH & HO.NO_2 & H_2.C.O.NO_2 & \\ \quad | & & \quad | & \\ H.C.OH + & HO.NO_2 = & H.C.O.NO_2 & + 3H_2O \\ \quad | & & \quad | & \\ H_2.C.OH & HO.NO_2 & H_2.C.O.NO_2 & \end{array}$$

A batch process is generally used with a nitrating acid of the composition 40% HNO_3, 58% H_2SO_4, 2% H_2O.

The nitration is carried out in a lead 'nitrator-separator' (equipped with water-cooling coils, air agitation and suitable feed and product pipes), at a temperature of 20—25°C.

When the reaction is completed, the air supply is stopped and the nitroglycerine phase separates as an upper layer, being eventually displaced by extra spent acid which is added at the base of the vessel, the liquid explosive then overflowing to a water-washing tank. A diagram of the plant is shown in Fig. 1.

When mixed with kieselguhr or other porous white earths, the product is known as dynamite.

A similar type of explosive, except that it is a solid, is the more recently discovered penta-erythritol tetranitrate (PETN), extensively used in the recent war. It is manufactured from formaldehyde and acetaldehyde by the Cannizzaro reaction:

$$CH_3CHO + 3HCHO = \{C.(CH_2OH)_3.CHO\}$$
$$\{C.(CH_2.OH)_3.CHO\} + HCHO + H_2O = C(CH_2OH)_4 + HCOOH$$
$$\underset{\text{penta erythritol}}{C.(CH_2.OH)_4} + 4HNO_3 = C.(CH_2OHO_2)_4 + 4H_2O.$$

(Lime is added to force the second reaction to completion, forming calcium formate.)

It is a crystalline solid, very convenient to handle, and is employed in fuses and also as a high explosive.

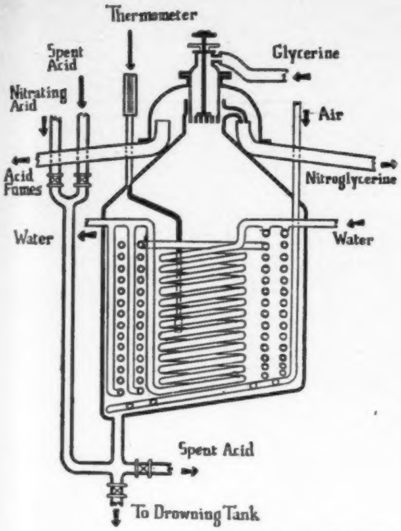
The famous Swede, Nobel, later used nitroglycerine in the manufacture of dynamite (1866) and blasting gelatine (1875). By this time other raw materials provided fresh developments. Nitration of cotton had been investigated, and by 1865 a nitro-cellulose had been produced by Sir Frederick Abel, capable of severe explosive qualities, and was termed gun-cotton.

Gun-cotton approximates to the formula: $C_6H_7O_2(O.NO_2)_3$ *i.e.* cellulose tri-nitrate (actually it is a mixed ester). A variety of manufacturing processes exist, a typical one being a batch nitration in iron or stoneware pots surrounded by cooling water and provided with fume exhaust lines. Again a mixed acid is used, with a higher H_2SO_4/HNO_3 ratio than with nitroglycerine. The nitrated product is subjected to various purification processes and is then partly dried until it reaches a moist condition. (Dry gun-cotton is too sensitive to shock.) The reaction may be formulated as:

$$C_6H_7O_2(OH)_3 + 3HONO_2 + H_2SO_4 = C_7H_7O_2(ONO_2)_3 + 3H_2O + H_2SO_4$$

The development of propellants proceeded from the discovery of nitroglycerine and nitrocellulose. The former is too unstable when pure, and the latter too rapid

*Reproduced, by permission, from *The Birmingham University Chemical Engineer*, 1950, 2 (1).



[*Courtesy Penguin Books Ltd.*

Fig. 1. Nitrator separator for nitroglycerine manufacture.

in combustion to be used singly as a propellant. Attempts were made to modify these explosives in such a way as to reduce their rate of combustion—mainly by adding cooling agents and inert materials. However, in the case of nitrocellulose the most satisfactory solution was found to lie in the application of gelatinising solvents such as an alcohol-ether mixture, which gave a horn-like product with a suitably-nitrated cellulose and was the first 'smokeless powder' (1886).

It was Nobel who made the greatest progress in the propellant field when he was able to gelatinise the fibrous gun-cotton with nitroglycerine, ultimately giving rise to the slow-burning propellant cordite—now 60 years old.

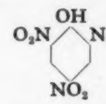
Cordite is manufactured by incorporating gun-cotton with nitroglycerine and adding acetone and mineral jelly. Sufficient acetone is added to dissolve *both* the explosives, the resulting 'dough' being transferred to presses and extruded through suitable dies to give threads or cords.

Solventless cordite arose from a serious acetone shortage in World War 1 and an efficient stabiliser and gelatiniser for nitroglycerine and gun-cotton mixtures was found in the compound diethyl diphenyl urea ('carbamite'), which becomes incorporated in the final product,[2] thus avoiding solvent recovery processes. Another development between the wars was the introduction of 'flashless propellants.' Many chemicals were examined before nitro-guanidine $H_2NC\ (:NH)\ NH.NO_2$ was chosen as the most effective agent in this connection.

High explosives

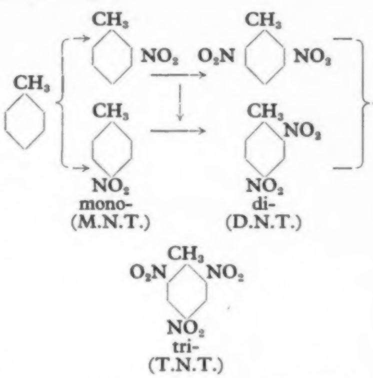
A third raw material, coal, began to provide explosive derivatives towards the end of the last century. The first of these was picric acid, obtained by the nitration of phenol and first detonated in 1871. Although extensively used in the 1914-18 war it has now given place to the superior T.N.T.

Picric acid or trinitro phenol



is no longer used because of the very sensitive nature of the metal salts formed.

Tri-nitro-toluene (T.N.T.) was the main high explosive used in World War 2. Again a mixed acid is used in nitration. Toluene is first nitrated to give the mono-derivative (mainly ortho- and para-substitution), which is then converted, either batchwise or continuously, to give tri-nitro-toluene:



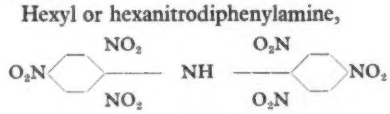
In the continuous process, M.N.T. and concentrated sulphuric acid are passed in opposite directions through a series of nitrator-separator pairs, the necessary nitric acid of various strengths being added to intermediate nitrator pots. Temperature control is important on such units and is obtained by the use of coils through which either steam or water can be passed. To avoid the separation of solid 'nitrobodies,' the temperature is increased towards the T.N.T. end of the plant, being about 82°C. at the final nitrator.[3]

Other non-symmetrical T.N.T. isomers are produced simultaneously and are removed by sulphite washing (converting all the T.N.T.s into soluble sulphonates). The product is finally washed, dried and flaked in a similar manner to that adopted for soap and caustic soda to give a product melting at 81°C.

For military use it is generally mixed with ammonium nitrate, owing to its own deficiency in oxygen. Because of its poisonous and dermatitic nature (common to most aromatic nitro compounds), much attention is given to such problems as dust control in all T.N.T. factories.

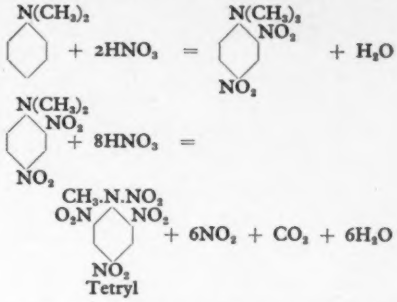
Aniline has provided two high explosives of note:

Hexyl or hexanitrodiphenylamine,



was favoured in Germany for use in mines and some types of bombs. Its very poisonous and highly dermatitic nature have limited its use in this country.

Tetryl or (2, 4, 6 tri nitro phenyl)—methyl nitramine is manufactured from dimethyl aniline *via* its soluble sulphate in a two-stage process,[4] in which the nitro groups are first attached to the phenyl ring:



Tetryl is used as a 'booster' for the main charge as, although stable when pure, it undergoes detonation fairly easily. It is extremely dermatitic. A 'booster' is generally an explosive of intermediate stability which picks up the explosive wave from the primary explosive, amplifies it and ensures complete detonation of the bursting charge.

Nitramines

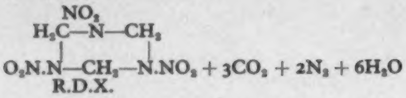
The major innovation in the high explosives field during the 1939-45 war was the considerable use of aliphatic nitramines, particularly cyclotrimethylene trinitramine, better known as R.D.X. This was the most powerful explosive used prior to the entry of atomic weapons. It is produced from coal, air and water in a remarkable series of reactions:

$$C + H_2O \longrightarrow CO + 2H_2 \longrightarrow CH_3OH \xrightarrow{O_2} HCHO$$

$$HCHO \xrightarrow{NH_3} \underset{\text{Hexamethylene tetramine}}{(CH_2)_6N_4} \xrightarrow{HNO_3} \underset{\text{R.D.X.}}{(CH_2)_3\,(N.NO_2)_3}$$

The final stage involves the nitrolysis of hexamine with 95 to 100% nitric acid.[5] Although R.D.X. was discovered as early as 1899 it was found impossible to manufacture it by a batch process, on account of the formation of large amounts of unstable by-products.[5] The latter were identified and a process of continuous nitration was devised, the by-products being safely destroyed by a hot-water dilution process, immediately following on the nitration. By this unusual step the process was rendered safe and practical. A unique feature is the use of 100% HNO_3 as the nitrating agent (H_2SO_4 attacks R.D.X.). Crystalline hexamine and concentrated nitric acid are continuously added to a cooled reaction mixture resulting from the same ingredients, the resulting solution overflowing to a dilutor which allows the R.D.X. to crystallise out and the dangerous by-products to decompose under controllable conditions.

$(CH_2)_6N_4 + 6HNO_3 =$



R.D.X.

The crude R.D.X. is milled to reduce crystal size and is purified by washing and boiling in water. R.D.X. is too sensitive to handle in the dry state, so it is normally mixed whilst still wet with suitable materials such as T.N.T. and aluminium to form explosive charges for shells, bombs and demolition charges.

Alternative methods of manufacture have been devised in America.[6] In Canada the initial reactants of the process used are formaldehyde, together with ammonium nitrate and acetic anhydride:

$$(1)\quad 3CH_2O + 3NH_4NO_3 + 6(CH_3CO)_2O = C_3H_6N_6O_6 + 12CH_3COOH$$

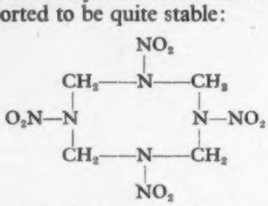
In the U.S.A. a 'combination' process has been evolved, giving 2 mols of R.D.X. per mol of hexamine, which is added to a mixture of nitric acid, ammonium nitrate and acetic anhydride. Thus reaction (1) above is combined with the simple nitrolysis of hexamine:

$$(2)\quad (CH_2)_6N_4 + 4HNO_3 = C_3H_6O_6N_6 + NH_4NO_3 + 3CH_2O$$

and the overall reaction is obtained[7] by adding equations (1) and (2):

$$C_6H_{12}N_4 + 4HNO_3 + 2NH_4NO_3 + 6(CH_3CO)_2O = 2C_3H_6O_6N_6 + 12CH_3COOH$$

By 1945, production had reached the extraordinary figure of 360 tons/day. The actual yield of the process is nearer 1.25 mols of R.D.X., as some 50 other by-products are also formed. Among these by-products is an interesting eight-membered-ring compound H.M.X.[8]—cyclotetramethylenetetranitramine—which is reported to be quite stable:



Other nitramine explosives developed in the U.S.A. included 'EDNA' (ethylene dinitramine),

$$\begin{array}{l} CH_2 \,.\, NH.NO_2 \\ | \\ CH_2 \,.\, NH.NO_2 \end{array}$$

which was obtained from ethylene urea and 'DINA' (di (2-nitroxyethyl) nitramine)

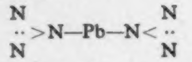
$$NO_2.\ N.\ (CH_2.\ CH_2.\ O.\ NO_2)_2$$

which was developed to meet the needs of a flashless explosive to prevent temporary blindness of naval fighing men in night warfare. It is prepared from ethanolamine $HO.CH_2CH_2.\ CH_2.\ NH_2$ by nitration.

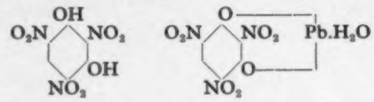
Detonators

This group of explosives consists mainly of freak compounds which are highly unstable and are made therefore in quite small quantities.

Mercury fulminate, C: N.O.Hg.O.N: C, was discovered in 1800 and is still in use, although lead azide



has superseded the former for many purposes. Another initiator is styphnic acid or tri-nitro-resorcinol



and its lead salt is frequently used in cap compositions.

Latest to be devised is the compound 'tetrazene,' or to give it is full title—guanylnitrosoaminoguanyltetrazene, with the strange structure

$$\begin{array}{l} ON.\ NH.\ NH.\ C.\ N : N.\ NH.\ NH.\ C.\ NH_2 \\ \qquad\qquad\quad \ddot{}\qquad\qquad\qquad\qquad\quad \ddot{} \\ \qquad\qquad NH \qquad\qquad\qquad\qquad NH \end{array}$$

Atomic explosives

For sheer destructive power all chemical explosives have been totally outmoded by the atomic explosives, uranium 235 and 233 and plutonium 239, which themselves may yet be surpassed by the speculative hydrogen bomb, which appears to result from deuterium-tritium interaction at exceedingly high temperatures of the order of 10^7 °C.[9]:

$$_1H^3 + {}_1H^2 \rightarrow {}_2He^4 + n + 17.6 \text{ mev.}$$

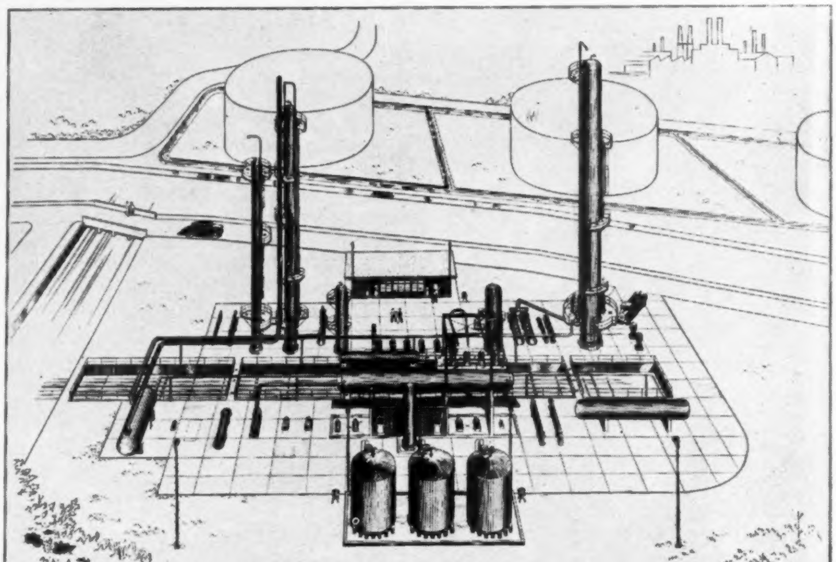
$$_1H^3 + {}_1H^3 \rightarrow {}_2He^4 + 2n + 11.4 \text{ mev.}$$

However, for most purposes, it seems likely that ordinary chemical explosives will be required for many years to come and will suffice most needs.

In the available space it has been impossible to mention more than a few aspects of explosives and many important fundamentals, and chemical engineering topics have been unavoidably omitted.

REFERENCES

[1] J. Read: 'Explosives,' 1942.
[2] J. N. Pring: *Ind. Chem.*, 1948, **24**, p. 467.
[3] T. Wallace: *Trans. Inst. Chem. Engrs.*, 1945, **23**, p. 193.
[4] R. N. Shreve: 'Chemical Process Industries,' McGraw Hill, 1945.
[5] W. H. Simmons, A. Forster and R. C. Bowden: *Ind. Chem.*, 1948, **24**, pp. 429, 530 and 593.
[6] 'Report of 110th A.C.S. Meeting,' *Chem. Eng. News*, 1946, **24**, p. 2467.
[7] E. R. Riegel: 'Industrial Chemistry,' Reinhold, New York, 1949.
[8] W. C. McCrone: *Anal. Chem.*, 1950, **22**, p. 1225.
[9] L. Ridenour: *Bull. Atom. Sci.*, 1950, **6**, p. 105.



NEW ALKYLATION UNIT

Nearly completed for the California Refining Co. at its Barber refinery in Perth Amboy, N.J., this 2,180 barrels/day sulphuric acid alkylation unit is scheduled to go on stream very soon, according to the builders, the M. W. Kellogg Co. Originally planned to help meet the 'octane challenge' of the higher compression engine, it is currently being somewhat modified to produce high-octane aviation petrol, in view of increased military requirements. The modification calls for the addition of a re-run tower for fractionating aklylate into heavy and light products. Not shown in this perspective view, this will be installed next to the debutaniser tower shown at the right of the picture. Primary advantage of this process alteration is to provide for a 'sharper' fractionation of the alkylate, an operation not considered essential to the production of quality motor petrol blending stock. When complete, the unit will be able to produce alkylate from a mixture of isobutane and either butylene or propylene. In the winter, when butylene is required for vapour-pressure blending in refinery petroleum, it will operate on isobutane and propylene, plus available butylene. At other times, the process will be normal butylene-isobutane alkylation.

CATALYSIS

Polymerisation processes

By S. L. Martin, M.Sc., F.R.I.C.

SINCE the last review[1] two important publications dealing with catalysis have appeared. In volume two of the annual series on 'Advances in Catalysis'[2] the previously defined policy of the editors has been maintained, in that eight authoritative articles on particular aspects of the subject by authors each a specialist in his field are presented. Of particular interest to the present report are articles on the mechanism of polymerisation of alkenes by Schmerling and Ipatieff, and on the use of hydrogen fluoride in various types of catalysis by Simons.

A report on the general discussion on heterogeneous catalysis, held by the Faraday Society in Liverpool last April, was published in September.[3] Contributions, by way of papers or discussions, were made by most of the leading workers in academic and industrial laboratories in all parts of the world. These contributions were largely concerned with the further application of the newer concepts and techniques indicated in the first review of this series, for example the 'patterning' of reacting molecules on to the catalyst surface, the electronic structure of reactant molecules and catalyst materials in relation to electron- and proton-transfer processes, and the use of radioactive isotopes as tracers. Much information of importance to the understanding of catalysts for hydrogenation, hydrocarbon cracking and the Fischer-Tropsch synthesis was presented, which will undoubtedly result in additional publications in the near future. For this reason and because the last two reviews have been largely concerned with heterogeneous catalysts of similar type, more detailed consideration of the results of that discussion will be left to a later review.

In this review, recent progress in the use and knowledge of the role of catalyst in the equally important processes of polymerisation is outlined. Synthetic polymer production has expanded considerably in the last 15 years. These polymers are now a major portion of the overall output of the chemical industries and of vital economic and strategic importance—as is evidenced by reference to high-octane fuels obtained by dimerising and trimerising low-carbon olefins in petroleum or natural gases; alkyd, phenolic and urea resins for the paint and adhesive industries and as materials and articles for building construction; other plastomers such as the polyvinyls, polythenes, polyurethanes and silicones with their wide range of application including that of synthetic fibres; and elastomers or synthetic rubbers such as the 'butalastics' (based on the monomer butadiene), polystyrenes and polychloroprenes.

The polymerisation process

The *addition* of a catalyst is by no means necessary in *all* polymerisation processes. Reference is frequently made to 'spontaneous' polymerisation, and it is well known that 'inhibitors' must be added to many monomers as commercially prepared to prevent the occurrence of this spontaneous process. On the other hand, some monomers, such as isobutylene and benzene, cannot normally be polymerised without the aid of a catalyst. The course of the polymerisation of a particular monomer/catalyst system may be profoundly altered by the addition of other substances, known as 'modifiers.' There is also evidence that with certain systems and conditions the presence of a 'co-catalyst' is necessary before the catalyst will function at all—in other words, the catalyst requires an 'activator.' It is a general fact that the conception of the purity of any chemical substance is based directly on the degree of refinement of the analytical procedures available, and as this improves, substances previously considered pure are often found to contain trace impurities in quantities sufficient to act as catalysts, inhibitors, modifiers or co-catalysts. It is thus quite possible that many so-called spontaneous polymerisations result from such trace impurities.

In the absence of concrete ideas on the polymerisation process, and in the light of the present realisation that polymerisation may occur by more than one mechanism, it was inevitable that many of the earlier prescriptions for catalytic processes should smack of cookery-book recipes. That the bulk of investigations prior to 1938 were largely empirical in nature, can be seen from the surveys of Staudinger and others summarised by Berkmann, Morrell and Egloff in 1940[4] and Lohse in 1946.[5] The best that could be done at that time was to list the various types of polymerisation processes according to the chemical type of monomer, to list the catalysts employed partly on chemical type, and indicate the lack of any comprehensive scheme. An indication of the range of materials suggested as catalysts for polymerisation processes is given in Table 1. (For butalastics alone, Marchionna[6] has indexed over 700 U.S. Patents and 300 substances mentioned as catalysts up to 1945.)

However, this earlier work produced enough empirical facts to provide the necessary impetus and guidance for the more detailed studies undertaken in the past ten years on the kinetics (mechanism and rates) of polymerisation processes. Notable success has been achieved and it is now possible to present established schemes for two, possibly three, mechanisms (which, however, do not apply to all known processes), to classify the various catalysts into an equal number of types and to offer an explanation for the action of modifiers and inhibitors. This has resulted from careful fundamental work with relatively simple systems, for example work on the reaction kinetics, effect of inhibitors and on co-polymerisation carried out by various schools in many countries. These schools have drawn heavily on other concurrent work such as that on the reactions of free radicals and the development of modern theories of acids and bases.

TABLE 1. CATALYSTS SUGGESTED FOR POLYMERISATION OF SOME ORGANIC CHEMICALS (from Berkmann *et al.*[4])

Organic monomer	*Polymer type*	*Catalysts*
1. Ethylene	Lubricating oils	BF_3 (1873), $AlCl_3$, H_2SO_4, $KHSO_4$, C, Al_2O_3, borax, silica gel, alkalis
	High-octanes	H_3PO_4, Co with Ag or Cu, benzoyl peroxide, ultra-violet light
	Low molecular weight fuels	Heavy metal sulphides and sulphates
2. Propylene	Lubricating oils	Clays, earths, BF_3
3. Butylenes	Fuels	$AlCl_3$, H_2SO_4, H_3PO_4
	Rubbers	Cu, Na metal, BF_3
4. Butadiene	Rubbers	HCOOH, Na-type metals, nitrogen bases, oxidising agents, $SnCl_4$, BF_3
5. Vinyl compounds	Rubbers	Peroxides and oxidising agents, metals (Na)
6. Acetylenes	Oils or low molecular weight compounds	Cu, Ni, Pt, $AlCl_3$, etc., charcoal, H_3PO_4, silica gel, ultra-violet light
7. Styrene	Polystyrenes	Metals, alkalis, O_2, benzoyl peroxide, $SnCl_4$, mercaptans, BF_3

The ideas associated with these last studies are basic to the new conception of the role of catalysts, and some reference to the main results and postulates is therefore necessary. Following that, the salient points concerning the general phenomenon of polymerisation are summarised. Finally specific types of catalysis are discussed with reference to selected systems.

Modern views on acids and bases[7, 8]

On the classical concepts an acid was considered as a substance containing replaceable hydrogen and capable in aqueous solution of producing by dissociation hydrogen ions (which were associated with solvent molecules to give positive ions of the type $(H_3O)^+$, and a base as one which produced hydroxyl anions $(OH)^-$. This accounted for the behaviour of the common acids and alkalis in water, but not for that in other solvents such as ethyl alcohol or chloroform, nor for that of bases such as the organic amines which contained no hydroxyl groups.

In the early 1920s two theories of acids and bases were formulated. The Brönsted-Lowry theory rapidly assumed prominence and was early applied to explain the general mechanism of acid-base catalysis in a variety of organic reactions such as isomerisation, condensation, decomposition and polymerisation. The Lewis theory at first did not receive much support, but has recently been strongly advocated, particularly in America, one of the reasons being that it provides an explanation for the action of such 'acid-type' catalysts as aluminium chloride and boron trifluoride which could not be fitted into the Brönsted-Lowry scheme of acids.

The Brönsted-Lowry or protolytic theory

According to this theory, an acid is a substance which tends to lose a *proton* (positively charged hydrogen nucleus or hydrogen ion), and a base one which tends to add a proton. This occurs independent of the nature of the solvent, and in solution there is not a large concentration of protons —in contrast to the classical theory which demands a high hydrogen-ion concentration for strong acids. Put simply, the acid-type A is in equilibrium with the corresponding base-type B and a proton H^+:

$$A_1 \rightleftharpoons B_1 + H^+$$

A and B being termed a conjugate acid-base pair. Typical acid-type species are hydrochloric, sulphuric, acetic and similar acids, negative ions such as the bisulphate HSO_4^-, and positive ions such as NH_4^+ and amino-ions $(RNH_3)^+$. Typical bases are ammonia and the amines, and negative anions of the weak acids such as the acetate ion $(CH_3COO)^-$ which tend to take up a proton giving the little ionised acid. In the general case, where A_1—B_1 and A_2—B_2 are two conjugate acid-base pairs, we have the following general equation and examples for ionisation in various solvents:

TABLE 2. RELATIVE STRENGTHS OF SOME ACIDS AND BASES (from Bell[7])

Acid	*Relative strength (ref. to $(H_3O)^+$)*	*Base*	*Relative strength (ref. to $(OH)^-$); (approx. values)*
$(H_3O)^+$ e.g. H_2SO_4 in water	55.5	$(OH)^-$ *e.g.* NaOH in water	10^{16}
Bisulphate ion	1.03×10^{-2}	Sulphide ion $S^=$	5×10^{14}
Oxalic acid	5.7×10^{-2}	Phosphate ion	10^{13}
Acetic acid	1.78×10^{-5}	Methylamine	7×10^{10}
		Acetate ion	5.5×10^{4}
		Ferrous ion in water $[Fe(H_2O)_5:OH]^{++}$	140
		Water	55

$$A_1 + B_2 \rightleftharpoons A_2 + B_1$$

$$H_2O + H_2O \rightleftharpoons [H_3O]^+ + (OH)^-$$

$$EtOH + EtOH \rightleftharpoons (EtOH_2)^+ + (OEt)^-$$

$$NH_3 + NH_3 \rightleftharpoons (NH_4)^+ + (NH_2)^-$$

$$CH_3COOH + H_2O \rightleftharpoons [H_3O]^+ + (CH_3COO)^-$$

$$H_2O + (CH_3COO)^- \rightleftharpoons CH_3COOH + (OH)^-$$

Dissociation then only occurs in those solvents capable of accepting a proton to give a complex ion, but nevertheless acid-base reactions such as indicator changes and titrations can be obtained in all solvents.

The general case of acid-base catalysis, which is known to occur in solvents other than water, can thus be conceived as a slow 'protolysis' in which the substrate behaves as a very weak conjugate base or acid and transfer of a proton from the catalyst to the substrate occurs. For simple systems the catalytic reaction rate constant is found to be a power function of the strength of the acid or base catalyst, the strength being expressed simply as the dissociation constant for an acid, or, for a base, as the reciprocal of the dissociation constant of the corresponding acid, allowance being made for the number of available protons from the acid and the number of equivalent positions at which the base can add a proton. In other words, the catalytic power of the acid or base is dependent on the proton-donating or proton-adding powers respectively.

Catalysts which are ionised completely in the solvent act entirely by virtue of solvent ions, for example with water the cation $(H_3O)^+$ is the acid catalyst and the anion $(OH)^-$ the base catalyst, irrespective of the particular acid or base. These are therefore the strongest catalysts of their class—hence the wide use of sulphuric acid and the caustic alkalis as catalysts. An explanation is also obtained for the (much milder) catalytic effect of acetic and oxalic acids which are practically un-ionised, the acid sulphate anion, and for that of the bases such as the phosphate ion, the acetate ion, methylamine and the ferrous ion. The strengths of these relative to the $(H_3O)^+$ and $(OH)^-$ ions are indicated in Table 2.

The Lewis theory

Substances such as the Friedel-Crafts catalysts (metal halides) cannot be fitted into the Brönsted-Lowry scheme, although in many reactions they behave as very strong acids. Lewis classified as acids all substances which can accept *electrons* from bases (*acceptor acids*) to form bonds of a co-valent type, and as bases all substance which can donate electrons (*donor bases*) for the formation of such bonds, irrespective of the chemical nature of the substances or of their exact mode of action. These donor bases must therefore have an unshared electron pair and will thus be able to accept a proton in the Brönsted-Lowry sense, so that there is no essential difference in the classification of bases under the two theories. Lewis's acceptor acids, however, must be substances in which the valency shell of electrons can be expanded by sharing one or more pairs of electrons. This is true of the metal halides normally used as catalysts, which Lewis classifies as 'primary acids,' but not of the typically strong common acids such as sulphuric acid. He therefore classifies these as 'secondary acids' which can be brought into his scheme only by postulating a complex formation with solvent molecules through a hydrogen bond. This constitutes the major difference between the theories, since Lewis's primary acids can only be brought into the Brönsted-Lowry scheme by postulating complex formation with another molecule which enables the complex to act as a proton donor, for example $BF_3.H_2O$.

The basic principle in the Lewis theory is the tendency for each atom in a molecule to complete its stable 'octet' of electrons, as exists in the stable rare gases, a tendency first indicated by Werner in the development of his co-ordination theory of complex formation in inorganic compounds. Thus boron and aluminium have three valency electrons, and in their trihalides share three others from the halogen atoms; as a result, the halogen atoms, each sharing one of the metal electrons, have a completed octet, but the metal atoms have only six electrons and are therefore capable of 'accepting' for sharing one electron pair, *i.e.* will share with another molecule having two unshared electrons or with two molecules each having one unshared electron. The basic substances have unshared electrons. For example, nitrogen has five valency electrons, and in ammonia three are shared each with a hydrogen atom (requiring only two electrons to complete its stable shell, not eight), thus leaving two unshared electrons. One molecule of boron trifluoride can therefore accept the two electrons capable of being donated by

one molecule of ammonia, to form a complex with co-ordinate co-valent or donor bonding. It is customary to consider that the atom donating the electrons will assume a positive charge (loss of negative charge) and that accepting them a negative charge in the complex molecule. The reaction is therefore depicted electronically as:

$$\begin{array}{c}\mathrm{H}\\ \ddot{}\\ \mathrm{H:\!N:}\\ \ddot{}\\ \mathrm{H}\end{array} + \begin{array}{c}\mathrm{F}\\ \ddot{}\\ \mathrm{B:F}\\ \ddot{}\\ \mathrm{F}\end{array} \longrightarrow \begin{array}{c}\mathrm{H\ \ F}\\ \ddot{}\ \ \ddot{}\\ \mathrm{H:N:B:F}\\ \ddot{}\ \ \ddot{}\\ \mathrm{H\ \ F}\end{array}$$

where, for simplicity, the electrons on the fluoride atom, irrelevant to the argument, are omitted or, omitting electrons entirely, as:

$$\begin{array}{ccc} & \mathrm{H} & \\ & | & \\ \mathrm{H-} & \mathrm{N} & \\ & | & \\ & \mathrm{H} & \end{array} + \begin{array}{cc} \mathrm{F} & \\ | & \\ \mathrm{B} & \mathrm{-F} \\ | & \\ \mathrm{F} & \end{array} \longrightarrow \begin{array}{ccccc} & \mathrm{H} & & \mathrm{F} & \\ & |^{+} & & |^{-} & \\ \mathrm{H-} & \mathrm{N} & \rightarrow & \mathrm{B} & \mathrm{-F} \\ & | & & | & \\ & \mathrm{H} & & \mathrm{F} & \end{array}$$

This tendency to accept electrons is stronger the lower the electropositivity of the metal atom and the greater the electronegativity of the non-metal in the compound, for example $BF_3 > BCl_3$, $BF_3 > AlF_3$, $AlF_3 > AlCl_3$. (In practice, boron trifluoride can be readily handled, but not aluminium trifluoride instead of which the chloride is therefore used.) The alkali and alkaline earth halides do not show this tendency to any appreciable degree. With the elements of the fourth group, such as titanium and tin, the mechanism is somewhat different; the co-ordination number is six, *i.e.* the elements can accept six pairs of electrons, thus giving them four over the stable octet, and these four are 'lost' to four halogen atoms, leaving a complex cation with four positive charges. This tendency will decrease from titanium to hafnium and from silicon to lead, and should be greater with silicon than titanium for example.

All these halides form well-known addition compounds with many substances, which are therefore classed as bases on this theory—for example, nitrogen compounds such as NO, NOCl, NH_3 and organic nitrogen compounds in which the N atom is the donor; similar phosphorus compounds, the P being the donor; sulphur-containing compounds such as H_2S, the sulphide ion, SCl_4, mercaptans, with the S atom as donor; and many oxygen compounds such as alcohols and ethers, an O atom being the donor. Other characteristics of these halides are the ease of hydrolysis, solubility in non-aqueous solvents, poor dissociation or ionisation in solution, and their tendency to form complex acids.

Acid-base catalysis in polymerisation processes

The tendency at present is to consider two broad initiation processes operative in particular systems or under certain conditions only:

(1) Proton transfer from acid catalyst to one carbon atom at a double bond of monomer molecule resulting in a negatively charged catalyst residue and a *carbonium ion*. The proton becomes bound by an electron which shifts from the other carbon atom at the double bond, leaving it deficient in an electron and thus carrying a positive charge as indicated below:

$$\mathrm{HA} + \begin{array}{c}\mathrm{R}\\ \mathrm{CH}\\ \|\\ \mathrm{CH}\\ \mathrm{R}\end{array} \longrightarrow \mathrm{A}^{-} + \begin{array}{c}\mathrm{R}\\ \mathrm{CH_2}\\ |\\ \oplus\mathrm{CH}\\ \mathrm{R}\end{array}$$

An example is the polymerisation of olefins by sulphuric acid. The corresponding mechanism of catalysis by bases would be transfer of a proton to the catalyst from the monomer molecule, leaving a molecule with one carbon atom carrying one extra electron, thus forming a *carbanion*.

(2) Electron sharing between the acceptor acid molecule and one carbon atom of the double bond, coupled with an electron shift from the other carbon atom, giving again a carbonium ion but one which contains the catalyst molecule, *e.g.*,

$$\mathrm{F_3B + CH_2{=}C}\begin{array}{l}\diagup \mathrm{R}\\ \diagdown \mathrm{R}\end{array} \rightarrow \mathrm{F_3\overset{\ominus}{B}-CH_2-C\oplus}\begin{array}{l}\diagup \mathrm{R}\\ \diagdown \mathrm{R}\end{array}$$

An example is found in the polymerisation of styrene with metal halides. Similarly, in catalysis by bases the monomer is the donor, the base the acceptor, and a carbanion results, but again containing a molecule of the catalyst. Thus if this process is operative, the resulting polymer should be found to contain catalyst residues, as has been demonstrated in certain cases such as the polymerisation of styrene by the sulphite radical.

Evans and Meadows[9] have, however, demonstrated that the low-temperature (-80°C.) polymerisation of *iso*butene by BF_3 to high molecular weight polymers requires a co-catalyst normally present in commercial *iso*butene and removable by rigorous purification. This suggests that the catalytic mechanism is not electron sharing as above, but proton donation from a catalyst/co-catalyst complex as has been postulated for the high-temperature polymerisation to low molecular weights. They point out that in no case is there direct evidence that a Friedel-Crafts catalyst acts independently of a co-catalyst, thus implying that all examples of this type of polymerisation catalysis may proceed according to proton transfer, as in scheme (1) above, through pre-formation of a Brönsted-Lowry type of acid from the halide and its co-catalyst as indicated below:

$$\mathrm{F_3B\underset{\mathbf{X}}{O}H + CH_2{=}C}\begin{array}{l}\diagup \mathrm{R}\\ \diagdown \mathrm{R}\end{array} \rightarrow \mathrm{F_3\underset{\mathbf{X}}{\overset{\ominus}{B}O} + CH_3-C\oplus}\begin{array}{l}\diagup \mathrm{R}\\ \diagdown \mathrm{R}\end{array}$$

Free radicals and their reactions[10, 11, 12]

A free radical results from homolytic fission generally of a carbon-hydrogen bond, leaving a radical with an unpaired electron at one of its atoms, generally a carbon atom, and thus with a greater reactivity than the parent molecule. The existence of free radicals was first demonstrated in 1929 by Paneth, who studied the methyl radical formed in the gaseous phase. The occurrence in solution of phenyl radicals was later suggested by Hey in 1934 to account for the decomposition of benzene diazoacetate in aromatic solvents; he conceived them as electrically neutral entities rather than ions. The importance of such radicals as initiators of reactions occurring by a chain mechanism was soon realised, and physicochemical methods were rapidly applied to their study, particularly in oxidation and polymerisation processes, which can now be interpreted in terms of the fundamental rate constants of the reactions.

Free radicals may result in one of three ways:

(1) By thermal reaction between two molecules leading to removal of a constituent part of the parent molecule or the formation of a di-radical (a single molecule with two unpaired electrons) as indicated below for styrene; a dash indicates the atom carrying the free electron:

$$\mathrm{2Ph.CH{:}CH_2 \rightarrow -CH_2.CHPh.CHPh.CH_2-}$$

$$\text{or } \mathrm{2Ph.CH{:}\ CH_2 \longrightarrow Ph.CH_2{-}CH_2{-} + Ph.CH{:}\ CH{-}}$$

Evidence based on the inhibition of polymerisation by *p*-benzoquinone, for example, favours the formation of a di-radical, since two of the monomer molecules appear to be added to one inhibitor molecule.[12]

(2) By the action of light, visible or ultraviolet, the parent molecule absorbing a quantum of energy which produces a di-radical or two mono-radicals as indicated below for styrene and benzoyl peroxide respectively:

$$\mathrm{Ph.CH{:}\ CH_2} + h\nu \rightarrow \mathrm{-Ph.CH.CH_2-}$$

$$\mathrm{Ph.CO.O.O.OC.Ph} + h\nu \rightarrow \mathrm{2Ph.CO.O.-}$$

(3) By reaction with another free radical, either with the formation of an additional radical or by a transfer mechanism in which an inactive molecule is formed from the old radical, as indicated by:

$$\mathrm{R{-} + CH_2 = CH.Ph \longrightarrow R.CH_2.CH.Ph{-}}$$

$$\mathrm{R{-} + CH_2{=}CH.Ph \rightarrow RH + CH_2{=}CPh{-}}$$

The third method is general to all polymerisation growth processes and to the initiation by catalysts in the form of free radicals, the evidence being that the addition reaction occurs in most cases. Catalysts acting in this manner are the peroxides or hydroperoxides, oxygen and the other oxidation catalysts. The latter probably act by first forming peroxides with the monomer molecules, the peroxides then producing free radicals by one of the general processes, namely decomposition by heat or—as indicated above for benzoyl peroxide—by light, or by catalytic effects of certain substances (which therefore function somewhat as co-catalysts). Thus

traces of polyvalent metals in a lower valency state will promote the formation of hydroxyl free-radicals from hydrogen peroxide according to the scheme:

$$H\text{-}O\text{-}O\text{-}H + Fe^{++} \rightarrow (HO{:})^{-} + \text{-}O.H. + Fe^{+++}$$

With hydrogen peroxide it can be shown that the degree of polymerisation depends on the concentration of the metal ion present.

Much work has been done on the reactivity of free radicals occurring in polymerisation systems in relation to the propagation of the chain and its termination, to reactions with inhibitors, modifiers and solvent molecules, and to the effects of substituents in the free radical on its reactivity.[12, 13] This work primarily concerns the mechanism of polymerisation rather than that of initiation by the catalyst, and will not therefore be treated here. It should, however, be noted that the life of such free radicals in solution may be as long as 100 sec., although their concentration seldom exceeds 10^{-8} mol./l. for monomer concentrations even of 10 mol./l.[11]

Alkali metal catalysts

The third broad group of polymerisation catalysts comprise the alkali metals—of which sodium is mainly used—and their organo-metallic compounds such as amylsodium or phenylsodium. These are the most widely used catalysts and their reaction mechanism has been most carefully studied.[6] These studies elucidated the mechanism of polymerisation and the structure of the polymer molecules obtained from butadienes. It is possible to postulate two mechanisms for the initiation process—formation of a free radical or of a '*carbanion*' (one carbon atom of the molecule carrying one negative charge in excess of that required for neutrality). In both, the monomer molecule is considered to be first adsorbed on the catalyst surface where an activated complex capable of reacting with other monomer molecules is produced. Recent studies have shown that Grignard reagents (such as triphenylmethylsodium, which produces the negative triphenylmethyl ion) and sodium in liquid ammonia (giving the amide anion) are effective catalysts for polymerisation of methacrylonitrile, and somewhat less so for the methacrylates and styrene. This, coupled with the known tendency for cation-forming substances such as fluorene, ethyl aniline and carbon dioxide to inhibit the sodium-catalysed polymerisation of butadienes, supports the carbanion mechanism. Mayo and Walling[13] contend that the study of the composition of co-polymers produced with catalysts from the three broad groups provides conclusive evidence for the non-radical nature of sodium polymerisations. Some of the data given by these authors is presented in Table 3. The difference in the two mechanisms is indicated below,

TABLE 3. EFFECT OF CATALYST TYPE AND INITIATION PROCESS ON INITIAL CO-POLYMER COMPOSITIONS (Mayo and Walling[13])

Monomer pair	*Mol. % Composition*	*Mol. % first named monomer in product for catalysis by formation of*		
		Radicals (Peroxides)	*Carbonium (Halides)*	*Carbanions (Sodium)*
Styrene/methyl methacrylate		51	>99	<1
Styrene/2,5-dichlorostyrene		40	93	—
Styrene/p-methoxystyrene	50/50	54	< 1	—
Styrene/vinyl acetate		98.3	>90	—
Styrene/vinyl ethyl ether		>99	< 9	—
Styrene/butadiene	15/85	11	—	15-18
Methylmethacrylate/acrylonitrile	50/50	69	—	<10
Methylmethacylate/methacrylonitrile	50/50	5	—	22

Na– representing atomic sodium and R⊖ a negative ion:

Free radical:

$$Na\text{–} + CH_2{:}\,CH.CH{:}\,CH_2 \rightarrow \overset{+}{Na}\overset{-}{C}H_2.CH{:}\,CH.CH_2\text{–}$$

Carbanion:

$$R\ominus + CH_2{:}\,CH.CH{:}\,CH_2 \rightarrow R.CH_2.CH.CH{:}\,CH_2 \quad (\ominus)$$

Table 3 also shows the differences in the mechanisms with the three types of catalysts represented by metal halides (carbonium ion), alkali metals (carbonion) and peroxides (free radicals). Mayo and Walling further point out that the initial polymer produced from a 1:1 feed of styrene/methylmethacrylate is nearly pure polystyrene, nearly pure polymethacrylate, or the 1:1 co-polymer for the three groups respectively.

'Alfin' catalysts

Morton has recently reviewed[14] some 24 papers published by himself and co-workers since 1944 on a new group of sodium-based catalysts. In the course of work on polymerisation of butadiene with amylsodium it was found that in the presence of di-*iso*-propyl ether an extraordinarily rapid polymerisation occurred—the fastest known for butadiene—giving a polymer with a high proportion of 1, 4 addition (as against 1, 2 addition with ordinary sodium), of high molecular weight and relatively insoluble in benzene. The active catalyst was effectively a mixture of sodium *iso*-propoxide with allylsodium formed thus:

$$C_5H_{11}Na + \begin{matrix} Me_2CH \\ Me_2CH \end{matrix}\!\!>O \longrightarrow Me_2CHONa + CH_2 = CHCH_3 + C_5H_{12}$$

$$CH_2=CHCH_3 + C_5H_{11}Na \longrightarrow CH_2=CHCH_2Na + C_5H_{12}$$

Each component was inactive on its own. Other active combinations can be prepared, in general by first reacting amyl chloride with sodium, then reacting half of the resulting amylsodium with a secondary alcohol containing at least one methyl group, and finally reacting the other half of the amylsodium with an olefin which must have a —CH=CH—CH$_2$— group either in a chain or ring (as in toluene)—hence the name ALcohol-oleFIN. The product containing NaCl, $R_1CH(CH_3)ONa$ and $CH_2=CHCH(R_2)Na$ remains in a finely divided suspension indefinitely.

The best R_1/R_2 combinations were Me/H, n-C_3H_7/H, and Me/$PhCH_3$, all giving approximately the same high molecular weights. The polymerisation process shows many other distinctive features compared with ordinary sodium polymerisations, notably that the highest molecular weights of 1,000,000 to 1,500,000 are obtained with the fastest catalysts and that the intrinsic viscosity of the polymer is independent of the amount of catalyst used and is regulated solely by the catalyst. It is considered that the combination of Na, Cl, alkoxyl and alkenyl ions and groups form a co-ordination complex aggregate on which the monomer molecules are adsorbed, giving potential anions by electron transfer from the aggregate. The freed anions in the aggregate then react with other adsorbed molecules with accompanying electron shifts, as for ordinary sodium catalysis. However, because of the longer life of the adsorbed monomer molecule in an activated state it has more opportunity for reaction with other monomer molecules first.

The toughness of the resulting polymer is a disadvantage, and is thought to be due to impurities in the catalyst which encourage double-bond formation and cross linking after metallation. Attempts are being made to purify the catalyst and co-polymers with styrene are being investigated.

Low-temperature catalytic polymerisations

In general, after spontaneous, photochemical or catalytic initiation to give an active monomer in the form of a free radical, carbonium ion or carbanion, all polymerisations must involve the following steps:

(1) Propagation, or growth of the polymer chain by reaction of the active group with a monomer molecule to give a larger active group, and so on.

(2) Chain termination, by

(*a*) Reaction between two active polymer groups,

(*b*) A transfer mechanism with solvent

or impurity molecules or catalyst residues to give an inactive polymer unit and possibly a new active species capable of further chain growth, or

(*c*) Reaction with a monomer molecule to give an inactive polymer unit and a new active species.

The function of the catalyst after initiation is to provide sufficient active species to counterbalance loss by any of the above processes and so continue the polymerisation at the desired rate. It is generally found that, other things being equal, the faster catalytic process gives polymers of lower molecular weight. Also, high temperatures, while increasing rates of polymerisation tend to give low molecular weight polymers. The rate of the composite process and the chain length of the polymer depend on the rates of the individual steps indicated above. A knowledge of the respective rate constants and of the energies of activation for the individual steps would permit a better choice of conditions to be made in order to obtain uniform polymers of desired chain length. Much progress is being made in this direction,[12, 13] but, as pointed out earlier, the results are beyond the scope of this article. It is, however, true to say that the synthetic polymers still fall short of the natural products such as cellulose, rubber, hair and other protein macromolecular substances, produced in plants and animals at atmospheric pressure and body temperature by relatively slow photo-catalysis or enzyme catalysis.

In this respect, reference has already been made to the low-temperature polymerisation of *iso*-butene with BF_3 catalysts to give high molecular weight polymers—of the order of 300,000. Another example is the low-temperature (0 to 5°C.) copolymerisation of butadiene and styrene to give the so-called 'cold rubbers' with improved elastic properties.[15, 16, 17] This process, originally developed in Germany, utilises a 'Redox' catalyst system as activator or initiator, consisting of an oxygen-rich compound such as benzoyl peroxide, persulphates or hydrogen peroxide, and a reducing medium such as metal sulphites, dithionites, amines or formaldehyde.

It is well known that the emulsion polymerisation of styrene and other vinyl compounds is very susceptible to atmospheric oxygen (even though peroxide or persulphate catalysts are used) which inhibits polymerisation, giving low molecular weights. Kern therefore added sulphites to act as anti-oxidants and unexpectedly obtained much faster rates. This was explained as catalysis by mono-acid radicals (anions) or free-radical ions produced by oxidation of the sulphite ions by the dissolved oxygen, and led to the development of Redox catalyst systems. Sulley has shown that styrene itself can be readily polymerised at 60 to 90°C. with sulphite containing 10 p.p.m. of cuprous ion according to the following suggested chain mechanism:

$$4Cu^+ + 2H_2O + O_2 \rightarrow 4Cu^{++} + 4(OH)^-$$

$$4Cu^{++} + 4SO''_3 \rightarrow 4Cu^+ + 4(SO_3^-)^\star$$

$$Ph.CH:CH_2 + (SO_3^-)^\star \longrightarrow Ph.CH_2.CH.(SO_3^-)^\star$$

Hobson and D'iami[17] have recently given data on the effect of traces of oxygen (added by dosage to an oxygen-free system) on the conversion rates in Redox-catalysed butadiene/styrene systems, which are now used for the major part of the cold rubber production. Such an effect was first indicated by the occurrence of slow irreproducible rates in a pilot plant operating at 41°F. with a typical Redox system of 0.1% cumene hydroperoxide, 0.1% ferrous sulphate, 0.15% potassium pyrophosphate and 1% dextrose as an antioxidant. It was found that as little as 5 ml. oxygen/100 g. monomer caused induction periods and reduced rates, presumably due to oxidation of ferrous ions to ferric ions resulting in a reduced activator concentration. Other Redox systems such as tetramethylene pentamine with cumene hydroperoxide or mercaptans with potassium ferricyanide were affected to a much smaller extent. An interesting suggestion in this respect is that by continuously measuring and regulating the amount of catalyst present in a polymerisation system with suitable oxidation-reduction electrodes and a potentiometric controller, good control of viscosity, molecular weight and solubility of polyvinyls may be achieved.[18]

Finally, the possible interpretation of these Redox systems in terms of catalyst/co-catalyst systems or *vice versa* and their probable relation to the natural enzymic processes are worth noting.

REFERENCES

[1] Martin: INTERNATIONAL CHEMICAL ENGINEERING, 1950, **31**, p. 213.
[2] 'Advances in Catalysis and Related Subjects,' Vol. 2, Academic Press, New York, 1949.
[3] 'Heterogeneous Catalysis,' Discussions of the Faraday Society, 1950, No. 8.
[4] Berkmann, Morrell and Egloff: 'Catalysis,' Reinhold, New York, 1940.
[5] Lohse: 'Catalytic Chemistry,' Chemical Publishing Co., New York, 1946.
[6] Marchionna: 'Butalastic Polymers,' Reinhold, New York, 1946.
[7] Bell: 'Acid-Base Catalysis,' O.U.P., London, 1941.
[8] Bell: 'Acids and Bases,' *Quart. Rev.*, 1947, **1**, p. 113.
[9] Evans and Meadows: *Trans. Faraday Soc.*, 1950, **46**, p. 327.
[10] Bawn: *J. Chem. Soc.*, 1949, p. 1042.
[11] Melville: *Endeavour*, 1950, **9**, p. 98.
[12] Burnett: 'Radical Polymerisation Reactions,' *Quart. Rev.*, 1950, **4**, p. 292.
[13] Mayo and Walling: 'Co-polymerisation,' *Chem. Reviews*, 1950, **46**, p. 191.
[14] Morton: *Ind. Eng. Chem.*, 1950, **42**, p. 1488.
[15] Sulley: *J. Chem. Soc.*, 1950, p. 1488.
[16] Kern: *Makromol. chemie*, 1948, **1**, pp. 199, 210.
[17] Hobson and D'iami: *Ind. Eng. Chem.*, 1950, **42**, p. 1572.
[18] U.S. Pat. 2,497,828.

EVAPORATION

Performances of evaporators, new uses, calculations, scale and corrosion

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THIS review of recent contributions in the field of evaporation is divided into three main sections: Performance of installations; trends in utilisation of various types; and theoretical aspects of evaporator calculations. Reference is also made to the auxiliaries of evaporator installations such as vacuum-producing equipment, condensers and control systems and, finally, to the ever-present problem of the control of scale. While no entirely new system of design has been introduced, the climbing-film type seems to be gaining in favour, owing to the short time of contact of the liquor with the hot surface (a matter of seconds), the small temperature difference required and the relatively more convenient mechanical construction.

Long-tube evaporators

A paper by Sawyer, Holzer and McGlothlin[1] describes the use of the long-tube unit for the concentration of black liquor in the kraft paper industry of America. This paper is a general review of the kraft process, but includes a heat and material balance sheet for the sextuple backward-feed evaporator unit. This unit concentrates the liquor from about 17.5% solids to 53%, with an evaporation of 130,000 lb./hr. of water, using 26,000 lb. of steam to give an overall economy of 5. Each evaporator contains 552 tubes of 2 in. i.d. and 24 ft. long in a steam shell of 4 ft. diam. and a dome diameter of 8 ft. The unit is fitted with a single surface condenser containing 1,146 tubes each 13 ft. long and 1 in. o.d. The vacuum is obtained from a two-stage steam-jet ejector.

The liquor with 53% solids is too viscous for further concentration in a film evaporator and is further concentrated in two large disc units each 8½ ft. diam. with 33 discs spaced 2⅝ in. apart. These discs pick up the liquor and evaporation is effected by passing in hot gases at 380°F. over the discs in the drum. This effects concentration to 64% solids. The combination of the two types of unit illustrates the advantages and limitations of the long-tube

type and the whole unit is clearly a good example of a careful technical approach to the problem. It is felt that the advantages of backward-feed systems might be considered more frequently.

The use of the long-tube Kestner evaporator in the cane sugar industry is described in a paper by Tromp.[2] At Casa Grande in Peru there are nine Kestners with 25-ft. tubes, each having a surface of 6,500 sq. ft., operating with 30 lb. of exhaust steam. These supply vapour to a group of final vacuum pans and also to the first effects of two quadruple-effect units. This represents a large evaporator installation and the steam utilisation and heat economy are worthy of examination. A further reference to the long-tube type, also for sugar processing, is given by McAlister[3]; the physical dimensions of the unit are given in some detail.

Film-type units

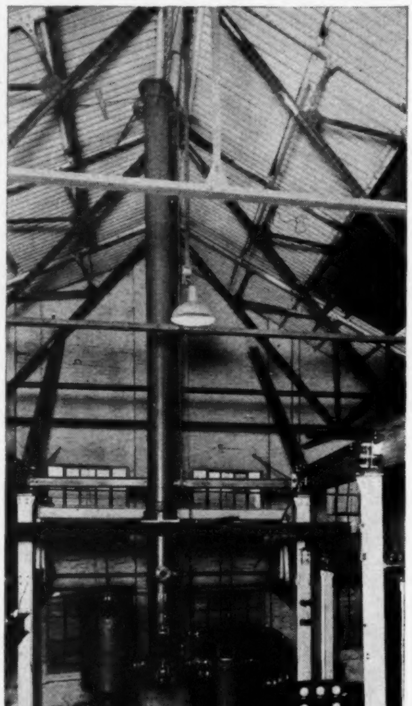
The attention given to the climbing film unit has tended to obscure the flexibility of the film-type system. Thus, film-type evaporators may have climbing, falling, or climbing and falling films. The falling film unit requires very small temperature differences and may therefore prove suitable for heat-sensitive materials, since the heating surface may be at the minimum temperature. This type has recently been installed[4] for the concentration of blood serum and gelatine. The climbing and falling type, with all the tubes inside the same steam shell, provides a very economical method for achieving a higher percentage evaporation of the feed than is obtainable with a single-pass climbing unit, but will usually offer a greater pressure drop. Fig. 1 shows a recently completed climbing and falling unit for concentrating confectionery liquors at atmospheric pressure. The preheater is seen at the bottom right-hand side, the main evaporating unit in the centre and the separator at the bottom left-hand side. This provides a very simple system for erection, since there are no parts to be mounted at the top of the unit. For vacuum work this will necessitate a low-level vacuum unit.

Fig. 2 illustrates the arrangement of a falling film unit installed for the concentration of a liver extract. In this plant the water is evaporated at 0.7 in. Hg absolute pressure, and the vapour produced is compressed in a thermocompressor to give a heating steam at 1.75 in. Hg pressure, thus giving a temperature difference across the heating surface of 28°F. In this installation the liquor is recirculated by pumping to the top of the tubes, over which it is distributed to form a film. The unit gives some indication of the difficult conditions encountered in the pharmaceutical industry. The problem of assessing the desirability and extent of recirculation has not yet been clearly analysed.

Where a single pass of the climbing film is insufficient to effect the required concentration then multiple circulation units may be used in which the vapour and liquid are separated at the top of each pass. In this form the first pass will have the largest number of tubes and the succeeding passes a smaller number. All the tubes are again inside one steam shell, which gives a compact assembly. This form of unit, for instance, may be used for concentrating fruit juices.

Scroll-type pans

Lyle[5] has reviewed heavy boiling in sugar concentration during the past ten years at a London refinery in which scroll-type pans are used. Although these pans have given very good service, it is reported that the scrolls have moved in time. The small heating surface of about 1 sq.ft./cu.ft. of mix is a drawback in this type of heater, when one would like twice this value. Lyle suggests that carefully designed circular ribbon-type heaters might be an improvement. As an illustration of the transfer coefficient obtained, he quotes one test in which the value was about 170 B.Th.U./hr./ft.^{2}/°F. at the start and fell off to 110 in 20 min. and down to 105 in 105 min. The practice of heavy boiling is considered an improvement, particularly as it reduces the number of boilings and simplifies the subsidiary equipment such as storage tanks, pumps, etc. At the same time, these heavy concentrates require expensive special pans. He emphasises the desirability of vacuum control by instrumentation on this process and illustrates with a chart the change in vacuum with time. Incidentally, it was found that the pan men were, in fact, performing the control very well before the control was put on. The control as originally applied was found to be incorrect, and he points out that it is desirable to know exactly what is wanted before leaving a control to do the job.



[Photo: Kestner Evaporator & Engineering Co. Ltd.

Fig. 1. Modern climbing and falling evaporating unit for concentrating confectionery liquors at atmospheric pressure. Bottom right can be seen the preheater, centre, the main evaporating unit, and bottom left, the separator.

Vacuum pans

The problem of providing a good heating surface for thick sugar liquors is discussed in a review of modern vacuum pans for the sugar industry by Venton,[6] who gives some comparisons in performance of several pans. He cites two modern forms of coil pan, one of 6,000 gal. capacity of 12 ft. diam., with a 5 in. o.d. coil flattened to $5\frac{9}{16} \times 4$ in., and the other of 5,000 gal. capacity, with 5 in. o.d. coil flattened to $6\frac{1}{8} \times 3$ in. These flattened coils are said to give reduced resistance to flow of the massecuite and hence better circulation. It is also easier to get a generous well in the centre, and the clearances between the coils are easier to arrange. These modern coil pans have much shorter coils than the old ones. Thus, while the older pans had coils of $2\frac{1}{2}$ turns, one of these newer pans has coils in the form of half circles flanged at the ends to give two manifolds, one for steam entry and the other for condensate outlet. The other new pan has a coil of $1\frac{7}{8}$ turns. These shorter lengths give better and more certain removal of condensate.

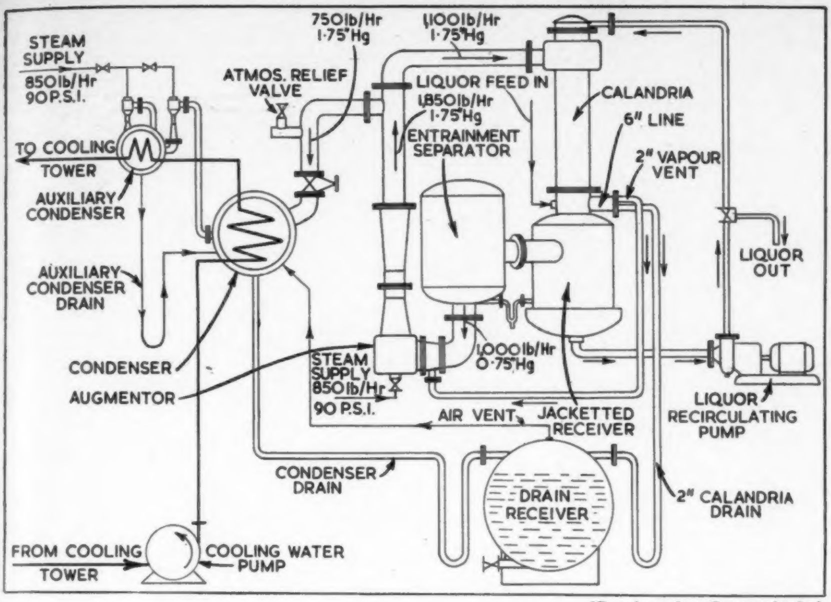
Webre[7] describes a vacuum pan with a central-top-driven agitator which is claimed to offer an improvement in heat transfer for sugar syrups.

Solar evaporation of salt

The production of Glauber's salt from Lake Chaplin is described by Holland.[8] A modern installation utilising solar heat for evaporation is used. The brine is pumped out in hot weather to 1,000,000 sq. ft. reservoirs, 10 to 15 ft. high, where the salts are deposited. In November the weak mother liquor is drained off and the salts stacked mechanically. These salts are then taken by conveyor to Holland evaporators (10 × 12 × 14 ft.), where they melt in their own water of crystallisation and are dried by passing in furnace gases at 1,500 to 1,700°F., which leave at about 160°F.

Concentration of caustic and sugar

In a British patent, Hedley *et al.*[9] describe a unit for concentrating solutions of calcium chloride or caustic where an anhydrous product is required. For calcium



[*Drawing: Apex Construction Ltd.*

Fig. 2. Arrangement of falling-film recompression evaporator for the concentration of a liver extract.

chloride the solution is concentrated to 60 to 75% and heated to 120°C. It is then sprayed into a unit with hot gas with a partial pressure of water less than 400 mm.

Coons[10] describes the design and operation of a four-effect vertical-tube evaporator for concentrating sugar solutions at positive pressures, the liquor having temperatures from 197 to 267°F. This paper outlines some of the advantages of pressure operation, but there have been few references to this form of operation.

Thermocompression

With the increasing costs of fuel the advantages of thermocompression are receiving more attention. Bardoux[11] gives figures on the coal utilisation per ton of sugar in the West Indies with and without thermocompression. The fitting of thermocompression increased the steam economy from 50 kg. of coal/ton of sugar to 40 kg., using a power-driven compressor. Turbocompressors and thermocompressors are discussed by Choquet[12] and Tromp has discussed the thermodynamics and economics of thermocompression in two papers.[13, 14]

Theoretical and experimental

Despite the increasing use of the film-type unit, there is as yet no really suitable method for computing the performance of a unit on a given liquor. Rumford[15] presented some data on the boiling of water, ethyl alcohol, toluene and nitric acid in a 9-ft. tube of ½ in. diam., using temperature differences of 18 to 41°F. This gave coefficients for water of 500 to 700, for alcohol of 300 and for nitric acid of 125 in B.Th.U./hr./ft.²/°F., based on the total area of the tube. The coefficients increased with higher feed temperatures, but attempts to divide the tube into boiling and non-boiling sections by using a travelling thermocouple to locate the point of maximum temperature were not entirely satisfactory, as this gave coefficients as high as 12,000 for the boiling section. These figures are clearly too high, but could not be explained. It was stated that if more than 10% of the feed was evaporated then the coefficient remained constant for the range in mass velocities covered. The work was not really undertaken as research on evaporator systems but to get some figures for boiling in long tubes, and insufficient details are given to form a true picture of what was happening in the tube.

In a recent paper, Badger and Pye[16] give a most interesting account of the development from a small-scale unit to a commercial one of a long-tube, once-through natural circulation unit for concentrating caustic soda solutions to final products of 96 to 97% caustic. The problem of corrosion of nickel and iron in the presence of chlorates is discussed at length and the many difficulties described in detail. The unit is heated by *Dowtherm* and the small one had seven tubes, ⅞ in. o.d. and 16 gauge of 15-ft. length in a vapour chest of 5-in. steel pipe. The overall coefficients on this unit ranged from 80 to 115. The full-scale plant consists of 20-ft. nickel tubes, ⅞ in. o.d., and is operated at 15 in. vacuum; it gives over 100 tons/day of 99% solids. The *Dowtherm* vapour is supplied at 700 to 710°F., which is a very high temperature for this form of heating. It is interesting to note that for vacuum greater than 15 in. the freezing point is greater than the boiling point from about 89 to 95%; hence some caustic might settle out.

McDonald and Rodgers[17] have presented an empirical equation for estimating the transfer coefficient for sugar concentration in the form:

$$U = 14.5 \frac{\left(\frac{t_j}{100}\right)^2}{\sqrt{\mu_j . c_j}}$$

where U is the overall heat transfer coefficient in kg.cal./m².min./°C., t_j is the boiling point of the sugar in °C., μ_j is the viscosity of the sugar in centipoises, and c_j is the specific heat of the sugar at the discharge temperature and concentration in kg.cal./kg. They have illustrated the use of this equation for finding the necessary heat transfer area in a later paper.[18]

Calculations

One or two papers have been presented with a view to simplifying the rigorous and rather tedious method for determining the temperature distribution and areas in multiple-effect systems. Coates[19] presents a method which is really based on expanding the simple rate equation to the form:

Total heat transferred =
Average latent heat × total evaporation or

$$\Sigma q = \lambda_{av} \Sigma E$$

Then if U_{av} be the average heat transfer coefficient and $\Sigma \triangle t$ the total temperature difference,

$$\lambda_{av} \Sigma E = U_{av} \Sigma \triangle t \Sigma A,$$

from which the total area ΣA can be obtained. The average latent heat λ_{av} is really defined by the relation:

$$\lambda_{av} = \frac{\text{Total heat transferred}}{\text{Total evaporation}}$$

but this is slightly modified to an empirical relation. Again the average transfer coefficient is given by:

$$\frac{1}{U_{av}} = \frac{1}{U_1} + \frac{1}{U_2} + \frac{1}{U_3}$$

where U_1, U_2, U_3, etc., are the individual overall transfer coefficients for the individual units. This method is illustrated by examples, and it is believed that the results are usually within 2% of those obtained by the more rigorous methods. It must be remembered that this method is unsuitable for those cases where steam is bled from the vapour space of one or more effects.

Storrow[20] presented a simplified method which is also unsuitable for those cases where there is appreciable flashing between the units or where there is bleeding of the vapours. A geometric method has been presented by Williams.[21]

Whilst these various methods are, no doubt, of use in estimating areas and in forming a quick picture of the effect of changing the conditions or the liquor, engineers must be prepared to carry out rigorous calculations of the Hausbrand type. A plant of the value of a large evaporator is surely worth the time required to solve a series of equations, and the idea that such vital aspects of design as areas and temperature conditions can

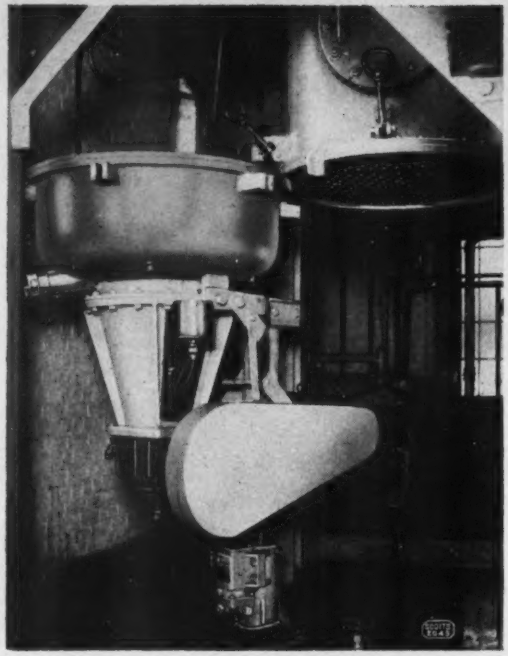


Fig. 3. Modern forced-circulation evaporating unit. The lower portion containing the driving mechanism can be easily swung away from the tubes for examination of the tubes and of the drive.

Fig. 4. Internal turbine-type circulating unit for evaporator.

[Photos: George Scott & Son Ltd.

be roughly estimated is a poor one. It is particularly unfortunate that more coefficients obtained in practice are not presented, since a large amount of practical data is wanted to form a practical correlation of transfer coefficients.

Bartholomew[22] describes in some detail the construction and operation of a small all-glass long-tube evaporator for laboratory use. This was 153 cm. long with an inside tube of 15 mm. and an outside tube of 30 mm. Using steam, he gave a capacity of 3.6 l./hr. of water under a pressure of 75 to 100 mm. Ethanol was evaporated at 8 l./hr., and the maximum temperature occurred at 4 to 5 in. up the tube.

Accessories

Webre[23] describes the essentials of a counter-current condenser and shows how the capacity of the vacuum system may be determined. Some of the pitfalls to be avoided in the use of vacuum equipment are discussed by Griffiths.[24] This paper gives a critical examination of the vapour line from the evaporator to the condenser, and illustrates the desirability of a genuine theoretical approach to a problem as opposed to the quick estimation methods. Instrumentation of evaporator units is discussed by Camp and Slater,[25] who stress the need for control of liquid level, product density and steam supply. Some calculations on reciprocating vacuum pumps are given by Hicks.[26] It is useful, perhaps, to note that steam jet ejectors are becoming increasingly used for the production of vacuum in evaporator plants, and that the virtues of positive-feed metering pumps for feeding the evaporator also offer advantages in obtaining good control of the plant.

Scale and corrosion

The problem of scale and corrosion constantly besets users of evaporators. This is examined by Hildebrandt and Warren[27] for calcium sulphate scale and for molasse stillage scale. The only cleaning method that gave reproducible results was hand polishing of the tubes with emery cloth. Chemical cleaning is discussed by Loucks and Groom[28] and the use of electric methods is illustrated by Springer,[29] who gives some details of the application of an electric potential to the tubes of an evaporator unit. His results are preliminary but are distinctly promising. The other method of tackling corrosion is the supply of new materials, with particular resistance to certain liquors. In this connection the construction of evaporators with *Keebush* for the body and shells and the use of carbon heating tubes offers distinct advantages in some industries.

Although no outstanding development has been claimed in the design of forced-circulation evaporators, it would be wrong to assume that they are not being actively developed. They are being installed for viscous liquids and also where the high velocity gives a short time of contact as with milk and juices.

Figs. 3 and 4 illustrate a modern forced-circulation unit. Fig. 4 shows the internal turbine-type circulating unit. In Fig. 3 it will be seen that the lower portion containing the driving mechanism can easily be swung away from the tubes for examination both of the tubes and of the drive.

REFERENCES

[1] F. G. Sawyer, W. F. Holzer and L. D. McGlothlin: *Ind. Eng. Chem.*, 1950, **42,** p. 756.
[2] L. A. Tromp: *Int. Sugar J.*, 1949, **51,** p. 272.
[3] McAlister: *Ibid.*, 1950, **52,** p. 273.
[4] Private communication.
[5] O. Lyle: *Int. Sugar J.*, 1949, **51,** p. 74.
[6] C. B. Venton: *Ibid.*, 1950, **52,** pp. 63, 96.
[7] A. L. Webre: *Ibid.*, 1949, **51,** p. 333.
[8] A. A. Holland: *Chem. Eng.*, 1948, **55** (12), p. 121, 1951 (1), p. 106.
[9] A. G. M. Hedley, *et al.*: Brit. Pat. 612,603 (1948).
[10] F. F. Coons: *Pro. Am. Soc. Sugar Beet Tech.*, 1948, **5,** p. 680.
[11] A. C. Bardoux: *J. fabr. sucre*, 1948, **89,** p. 126.
[12] A. J. Choquet: *Ibid.*, 1947, **88,** pp. 32, 52.
[13] L. A. Tromp: *Int. Sugar J.*, 1947, **49,** p. 289.
[14] *Idem.*: *Proc. Ass. Sugar Tech. Cuba*, 1947, **21,** p. 241.
[15] F. J. Rumford: *J. Soc. Chem. Ind.*, 1947, **66,** p. 309.
[16] W. L. Badger and D. J. Pye: *Chem. Eng. Progress*, 1950, **48,** p. 486.
[17] J. C. McDonald and T. Rodgers: *Int. Sugar J.*, 1947, **49,** p. 205.
[18] *Idem.*: *Ibid.*, 1947, **49,** p. 264.
[19] J. Coates: *Chem. Eng. Progress*, 1949, **45,** p. 25.
[20] J. A. Storrow: *Ind. Chemist*, 1948, **24,** p. 311.
[21] G. J. Williams: *J. fabr. sucre*, 1948, **89,** p. 147.
[22] W. H. Bartholomew: *Anal. Chem.*, 1949, **21,** p. 527.
[23] A. L. Webre: *Int. Sugar J.*, 1949, **51,** p. 224.
[24] H. Griffiths: *Trans. Inst. Chem. Eng.*, 1945, **23,** p. 113.
[25] A. C. Camp and L. E. Slater: *Chem. Eng.*, 1950, **57,** p. 108.
[26] T. G. Hicks: *Ibid.*, 1950, **57** (6), p. 108.
[27] F. M. Hildebrandt and K. H. Warren: *Ind. Eng. Chem.*, 1949, **41,** p. 754.
[28] Loucks and C. H. Groom: *Paper Trade J.*, 1948, **127** (25), p. 26.
[29] H. P. Springer: *Int. Sugar J.*, 1948, **50,** p. 242.

Mr. Alan Stubbs

We very much regret to have to announce the sudden death in January of Mr. Alan Stubbs, author of the article 'Modern Drying Plant,' which appeared in our February issue. This article was the first of a series which Mr. Stubbs was writing for us. Owing to his death this project must be abandoned.

Only 29 years old, Mr. Stubbs worked for the Kestner Evaporator & Engineering Co. Ltd. in the position of Laboratory Superintendent, being responsible for a wide range of chemical process work and plant development. His tragically early death is keenly felt by his employers and all who knew him.

'I.C.E.' April

This issue will include an article on the **Development of Coal and Coal Tar as Sources of Chemicals,** a survey of **Drying Technique,** a description of a **New Plant for Caustic and Chlorine Manufacture,** and an 'I.C.E. Review,' **Crystallisation,** besides our usual features.

Research on Building Materials

CEMENT, concrete, flooring materials, metals and paint are among the building materials under investigation by the Building Research Station of the Department of Scientific and Industrial Research. The progress of these studies is recorded in 'Building Research, 1949,' recently published by H.M. Stationery Office (3s.). In this report it is stated that a laboratory has been opened at Thorntonhall, near Glasgow, to study the particular techniques and building problems of the north and help in the development of indigenous resources for their manufacture.

Cement

Tests have been made on the effect on the degree of hydration of cement of three surface-active agents, two of which are used for air entrainment. The agents have been used in normal Portland cement pastes in the proportions recommended by the manufacturers and also in greater and less amounts. No significant difference was found between the degree of hydration of the control specimens without additions and those with different proportions of the surface-active agents.

British Standards for cements include a compressive strength test on mortar cubes compacted by means of a high-frequency vibration machine. As a result of the introduction of this test as a standard, a number of vibration machines were constructed. The troubles experienced with those machines which became defective were examined by the British Standards Institution Cement Committee, and a modified design was drawn up, together with a more rigid specification for the construction of future machines.

Concrete

The effect on reinforcement of the addition of calcium chloride to concrete has been examined for 1:6 mixes with different depths of cover to the reinforcement, and for periods of exposure up to one year. So far, the amount of corrosion is small, though there is a greater degree of corrosion with thin than with thick covers and more in those concretes containing calcium chloride than in those without.

After considerable delay in obtaining plant with the necessary degree of control, the programme of work on the steam curing of concrete proceeded. Data were first sought on the effect on ultimate strength of three variables, namely the initial curing period, the steam-curing period and the steam temperature.

Joint filling of floors

The filling of joints in food warehouse floors with bitumen and tar products has been investigated as a result of the considerable wastage of foodstuffs caused by the inroad of pests which find harbourage in cracks or joints in timber floors or at junctions with walls. The condition of these floors varies widely, the joints being sometimes irregular and wide, while the floor as a whole may move under traffic or loading. A mixture of bitumen, wax, rubber and a filler, suitable for hot application, was selected, the composition being adjusted to give the physical properties needed, according to the size of the joint to be filled. The technique for cleaning and filling the joints was found to have an important bearing on the success of the operation. Practical trials on warehouse floors are so far proving satisfactory.

Paints

In order to prevent the development of acidity with certain paints, the effect of using acid-absorbing pigments in the primer is now being investigated. Two putties were also designed to avoid the use of linseed oil, but neither was durable.

Metals

Investigations on the corrosion of aluminium and its alloy were continued. The effects of one test demonstrated the desirability of using a protective treatment, such as a bituminous paint, where aluminium or a susceptible alloy is to be placed in contact with mortar or plaster in circumstances where the conditions are liable to be damp.

Flooring materials

During the past few years the flow of requests for tests on flooring materials has reflected the continued search by manufacturers for suitable materials to cover concrete floors. Tests have been carried out on jointless, sheet and tiled materials, and it is interesting to note that sheet materials form by far the smallest number. Even these are not new types but modifications of rubber sheet, and it seems that a new substitute for linoleum has not yet appeared in commercial quantities. The only novel material in this group used polyvinyl acetate emulsion as the binder. None of the tiles tested was in use before the war, and all but one of them incorporated synthetic resin as the base. Those using thermoplastic resins have generally proved to be more successful and less restricted in use than those using thermosetting resins.

Chemical attack on chimneys

Mention was made in the report for 1948 of the pilot flue which has been constructed of heat-resisting glass to study flue conditions. During the year it has been used to study condensates. The results obtained so far suggest that chemical attack on chimneys used for domestic boilers, etc., cannot be accounted for by the formation of sulphuric acid, as has previously been assumed. A more probable mechanism is the production of a series of compounds of ammonia, sulphur dioxide and oxygen. An interesting conclusion is that damage may be most rapid when a flue which has been lined with cement or cement-lime mortar is out of use; liquefaction has been observed a few hours after the fire has been allowed to go out. A major factor in the wetting of brickwork and staining of plaster appears to be the use of wet fuel which can introduce more water than the wet rubbish. In the course of work a number of samples of coke were found to have a water content averaging 23%. Fuel may be delivered wet or become wet through insufficiently covered storage.

Full-scale trials on non-traditional types of flue materials have also been started. Asbestos cement, cast iron, wrought iron and three varieties of enamel on steel are being used, all attached to boilers of similar design.

Fire protection of buildings

Close collaboration with the Joint Fire Research Organisation has been maintained throughout 1949 and the station has assisted in a number of investigations. Further tests on prestressed concrete floor systems have shown that more than sufficient fire resistance can readily be obtained by providing appropriate thermal insulation for the concrete.

Preliminary consideration has been given to the wider aspects of the relation between structural design and fire-resistance requirements. Fire protection has usually been obtained by one or two methods: either the necessary fire resistance was inherent in the structure by virtue of structural requirements (for example, with brick walls), or fire protection was provided independently, as, for example, the concrete protection to structural steelwork. The development of more precise methods of structural design, especially in connection with structural frameworks, raises a need to correlate more closely the structural design of members with their fire resistance.

The question of the design of chimneys from the standpoint of fire risk has received attention, in order to meet the numerous requests for advice on the design of non-traditional forms of construction. Some use has been made of American data, but the variety of designs and lack of experimental results have caused some difficulties.

As a preliminary to undertaking the major task of preparing for the Codes of Practice Committee, a comprehensive Code on fire precautions in buildings, an officer of the station visited the U.S.A. and Canada. Much general information on American practice in fire protection was obtained and it seemed that, although this country was in no way behind as regards fundamental knowledge, there was greater progress in North America in the incorporation of the results of fire research in building regulations.

New Plant for Testing Pressure Vessels

A PULSATING pressure plant has recently been installed in the pressure vessel laboratory of the British Welding Research Association at Abington, near Cambridge, for testing vessels and other products by repeated applications of pressure.

The advantages of this type of test over the normal static pressure test are, firstly, that vessels which will be subjected to varying pressures in service can be tested under similar conditions during their initial development so that a relation between material properties, service stress and life expectation can be established and, secondly, that it will distinguish between two designs of otherwise equal merit far more readily than will a static test.

The simplest form of plant for this purpose would be a pump whose connection with the test vessel is alternately made and severed. Several designs of pulsating pressure plant are possible, the main difference being the manner in which the valve which controls this connection is operated. In the past this valve has been controlled either by a cam operated from the shaft of the main pump, or electrically operated over relays controlled by a pressure gauge having two contacts set at the desired pressure limits, or by various other means, all of which had attendant disadvantages.

In the plant which has been installed at Abington this valve is controlled by a pneumatic-hydraulic arrangement. The controlling mechanism consists of a piston travelling in a cylinder (marked 'servo piston' and 'servo cylinder' in Fig. 1), which is so designed that the piston will move when the pressure difference between its two sides is 1 to 1½%. To the space in the cylinder on one side of this piston is communicated the pressure in the test vessel, and to the other side that set in the 'upper-pressure-limit vessel.' As can be seen from Fig. 1, the main exhaust valve controls the passage of fluid from the test vessel to the large cylinder in which is set the lower pressure limit. When this valve is open, the test vessel and the 'lower-pressure-limit vessel' are in connection, and therefore the pressure in the test vessel is that of the lower limit vessel. When the valve is closed, the main pump draws fluid from the lower-pressure-limit vessel, and pumps this into the test vessel. As stated above, this pressure is communicated to one side of the servo piston, and when it reaches 1½% more than the upper pressure limit the piston travels along the cylinder. In so doing it moves the connecting lever which operates the two servo valves. These are attached to the lever in such a way that when one is open the other one is closed. Assuming that the test vessel has been pumped to its upper limit and the piston has travelled along its cylinder, the upper servo valve will be opened and a direct passage is available for fluid from the top of the exhaust valve to the suction side of the pump. Since now there is very little pressure above the exhaust valve and the full upper limiting pressure below it, this valve is forced open. As soon as this is done, fluid flows from the test vessel into the lower-pressure-limit vessel, and the pressure in the test vessel drops to that in the lower limit vessel. At the same time the pressure on one side of the servo piston drops to this lower limit, and therefore the piston travels in the reverse direction, closing the upper servo valve and opening the lower one. This permits fluid under pressure from the accumulator to pass to the top of the exhaust valve piston and, since the pressure on the top is now greater than that at the bottom, this valve closes and a fresh cycle of pumping up commences.

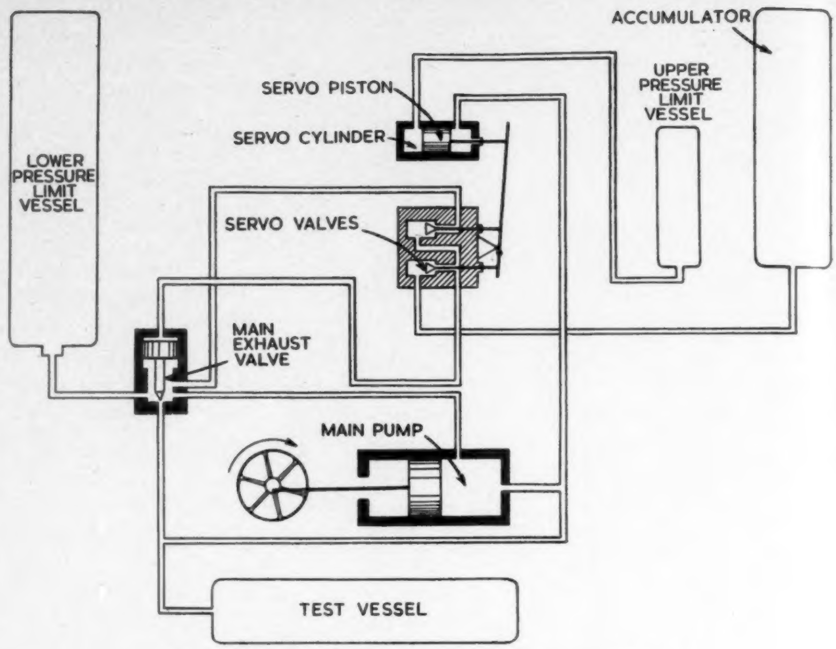


Fig. 1. Simplified layout of pulsating pressure plant.

The main pump, by means of which the test vessel is subjected to pressure, is a three-ram pump capable of a delivery of 12 gal./min. against a pressure of 6,000 lb./sq.in., and is driven by a 75-h.p. electric motor. In addition to this pump there is a small pump capable of a delivery of 1 gal./min. against a pressure of 6,000 lb./sq.in., and an air compressor with an intake of 40 cu.ft./min. These latter two are used for pumping air and water into the lower-pressure-limit vessel, the upper-pressure-limit vessel and the accumulator before the commencement of the test for setting the desired conditions.

The controlling valves, all of which have been omitted from the diagrammatic layout herewith, are so arranged that it is possible to vary the shape of the loading cycle and also to alter the limits of the test while this is actually proceeding. The upper and lower pressure limits between which pulsations can be obtained by the plant as erected at present are any two pressures between 0 and 6,000 lb./sq.in., and it is possible to obtain up to 100 cycles/min. in vessels of moderate size.

Recent publications

Multi-wall paper sacks. The manufacture of paper from timber to finished product, with special reference to the manufacture of multi-wall sacks, is described in 'The Medway Story,' published by Medway Paper Sacks Ltd. These sacks are used for packing chemicals, fertilisers, etc., and the many variations in proofing and design which can be made to meet individual requirements are outlined.

Steel pallets. The design, construction and uses of steel pallets are described and illustrated in a brochure entitled 'Flow,' published by Fisher & Ludlow Ltd. A series of photographs indicates the uses of steel pallets and other items of mechanical handling equipment.

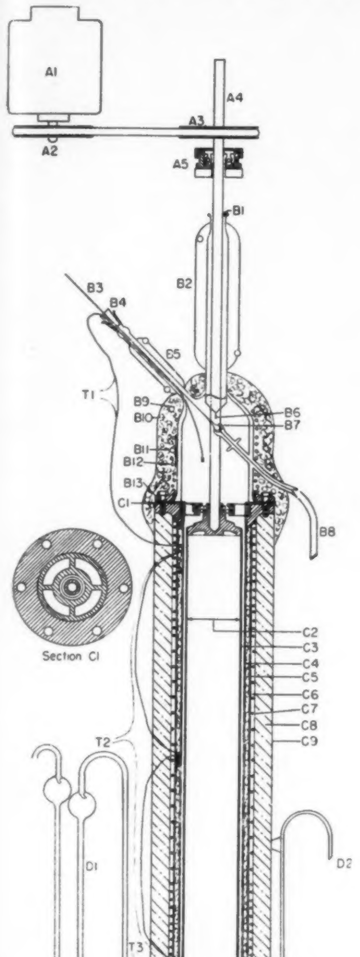
Organic chemicals. Current prices and other details of more than 1,800 organic chemicals, many not previously available to research workers, are given in a new catalogue issued by L. Light & Co. Ltd. This company is erecting new laboratories at Colnbrook, near Slough, and will be increasing their range of chemicals during the next two years.

New Fractional Distillation Column

A NEW rotary concentric-tube distillation column, having a very high efficiency factor for fractional distillation, has recently been developed at the U.S. National Bureau of Standards by C. B. Willingham, V. A. Sedlak, J. W. Westhaver and F. D. Rossini, in connection with the co-operative research programme on hydrocarbons jointly being carried out by the Bureau and the American Petroleum Institute.

Experimental programme

The present-day demands for fuels providing greater efficiency, higher speeds and more concentrated power, as well as the development of various devices making use of new types of hydrocarbons, has resulted in a transition in the petroleum industry from the manufacture of broad fractions towards the production of narrower fractions and even purer compounds. The experimental programme now under way at the U.S. National Bureau of Standards has two objectives: (1) The investigation of the chemical constituents of the crude oil based upon the actual isolation of pure hydrocarbons; and (2) the securing of high-purity standard samples for the calibration of instruments used in analysing such complex mixtures as aviation fuels and synthetic rubber components. The new column, which is expected to have wide application to fractional processes, will aid materially the Bureau's hydrocarbon research programme.



Schematic diagram of the new column.

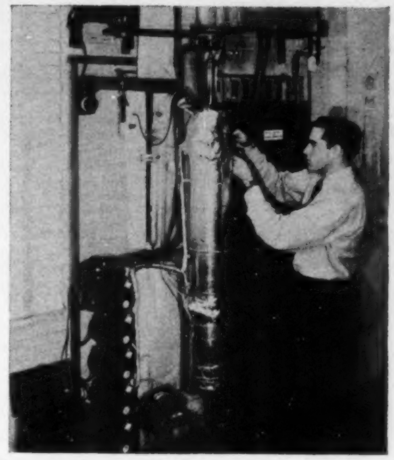
Numerous devices, based on both theory and experiment, are available for producing high separating efficiencies in distilling columns. Among these are open tubes of small diameter, concentric tubes with a small annular space, and parallel plates with a small space between them. All, however, are limited in use by a relatively low throughput—that is, quantity of material that may be volatilised per hour. According to theory, the efficiency may be improved by decreasing the spacing of the tubes or plates of the column, by decreasing the throughput, or by increasing the rate of diffusion of the gas molecules through the distilling column. Decrease in the spacing (or diameter) has already been carried about as far as practicable, while decreasing the throughput below the present low values is not feasible. Moreover, in a static apparatus, for a given temperature and composition, the rate of diffusion of the gaseous molecules is substantially constant.

Design of column

Accordingly, the new column was designed to improve separating efficiency by increasing the diffusion rate of the molecules in the gas phase. This is accomplished by forcing the gas into turbulence through rotation of the inner closed cylinder in a concentric-tube rectifying section.

The apparatus consists of three parts—the *Pyrex* head, steel rectifying section and *Pyrex* pot. All three sections are provided with external heating elements and copper-constantan thermocouples for temperature regulation.

The steel rectifying section of this column is the empty annular space, 0.043 in. (1.09 mm.) wide, formed by the inside surface of a stationary outer cylinder and the outside surface of a rotating closed inner cylinder, 2.928 in. (7.44 cm.) in outside diameter and 23 in. (58.4 cm.) in length. A motor-and-pulley system drives



Removing a test sample of distillate from the new rotary concentric-tube column in the distillation laboratory at the U.S. National Bureau of Standards.

the rotor at speeds up to 4,000 r.p.m. The outer cylinder is enclosed in an asbestos-covered metal heating jacket surrounded by three separate *Nichrome* heating elements for the top, middle and bottom portions of the jacket respectively. The heating elements are covered externally with magnesia insulation and a layer of aluminium foil.

The *Pyrex* glass head consists principally of a water-jacketed condenser directly above an opening into an electrically heated chamber, in which the liquid reflux may be collected and sampled by means of a glass valve. The heated chamber is surrounded by asbestos wool insulation and aluminium foil.

The pot, made of a 3-in. *Pyrex* pipe, is sealed at one end and provided with a butyl carbitol manometer and a tube for withdrawing samples. Heated externally by a *Glasscol* special sleeve-type heater, it is surrounded by magnesia insulation covered with aluminium foil.

Thermo-elements

Three thermo-elements are provided. One measures the difference between the temperature of the top portion of the rectifying section and the liquid-vapour equilibrium in the head. Another measures the difference between the temperature of the middle portion and the mean temperature of the top and bottom portions of the rectifying section. A third is used to determine the difference between the temperature of the bottom of the rectifying section and the temperature of the liquid in the pot.

For high values of throughput—2 to 4 l./hr. of liquid—this distillation column, when operated at 4,000 r.p.m., has an efficiency factor about ten times those previously reported for other rectifying columns.

New Plant and Equipment

Ceramic pressure filter

A new type of pressure filter has been designed by Doulton & Co. Ltd.: (*a*) To provide a large capacity unit, suitable for running at high pressures; (*b*) to reduce to a minimum the time required for cleaning and maintenance; and (*c*) to allow easy cleansing by back-flushing, adequate space for thick cake build-up and heavy sludge accumulation, and to provide for easy removal of the accumulated sludge.

Made from acid-resisting ceramic, the unit comprises 54 porous candles, 10 × 2 in., in banks of three on 18 perforated tube fittings. Each perforated tube can be removed through the top plate by slackening off two nuts. Adequate sealing is achieved when these nuts are thumb-tight or, at most, tightened up with a tommy bar. The three elements on a fitting can be brushed clean without removing them from the perforated tube and can be replaced immediately in position.

External piping and valves provide for back-flushing where this method of cleaning is adopted. The bottom of the unit slopes towards a sludge door, which is removed by the turn of a hand wheel and which is large enough to allow an arm to reach inside the casing.

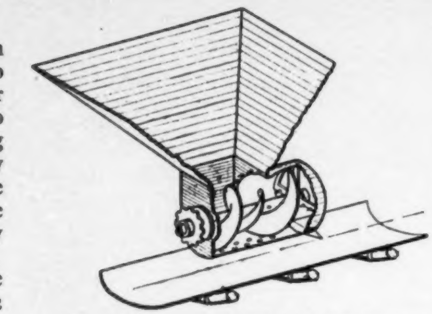
A pressure gauge is fitted on to the outlet chamber on the top front of the unit and will indicate when cleansing is advisable or give immediate warning of any failure. Air vents are provided for bleeding away any air trapped in the unit.

The number of 10-in. elements in the unit can be adjusted to multiples of 18 by increasing or decreasing the length of the perforated tube fittings and the length of the steel tube between top and bottom castings.

For water filtration for breweries, mineral water and similar works, the unit is supplied with top and bottom castings of best-quality gun-metal, all parts being hot-tinned or galvanised as a protective finish. It is anticipated that the unit will also be produced in the near future in stainless steel and other materials to enable it to be used where corrosive liquids are being processed.

Metal-working press

A recent accessory for the *Hydroram* repair kit introduced by Chamberlain Industries Ltd. consists of a 5-ton hydraulically operated press, fabricated from mild steel. A standard pump and ram unit can be fitted to provide the power source, and the press is suitable for handling all classes of light assembly work covering bending, straightening, clamping and shearing. Provision is made for fitting special tools to the work table. The base is provided with fixing holes for bolting the press to a work bench or table.



The sulphur crusher described below.

Sulphur crusher based on meat mincer principle

The Freeport Sulphur Co. built a portable crusher in 1944 that solved a problem that none of the crusher manufacturing companies were able to tackle successfully. Two of the crushers have now been in service for six years, handling a million tons/year, with a capacity of over 2,000 long tons/8-hr. shift. They reduce ton-size blocks of sulphur to 12 in. and also feed a conveyor system at the rate of 350 long tons/hr. per machine.

As described in the *Mining Engineer*, at Freeport Sulphur's loading operations at Grand Ecaille, Louisiana, liquid sulphur is pumped to two bin sites, and then cooled. The main conveyor is located between the two bins. On each side of this conveyor a travelling cross-conveyor is moved across the 200-ft. width of the bins as loading progresses. A portable 5-ton capacity hopper is mounted over the crusher-feeder. The hopper receives feed from a 2½-cu. yd. bucket, and moves across the cross-conveyor on rails as the shovel digs across the sulphur bin. From 1934 to 1944 lumps were caught on a grid with 12 by 18-in. openings, and two men broke the lumps of sulphur to conveyor size with picks. This was a costly and dangerous operation which suggested the use of a crusher. Most manufacturers shied away from a 350-ton/hr. portable crusher, and those who tried to work out something came up with more weight than the conveyor trusses could stand.

One day in 1943 it was noticed that sulphur samples were being crushed by a meat mincer, and this observation gave birth to the successful Freeport crusher. This crusher consists of a screw operating in a trough welded to the bottom of a hopper. The screw acts as a feeder to the conveyor belt, thus eliminating the usual pan feeder. Lumps too large to settle into the trough are broken off at the bottom by the screw ribbon until they settle on the screw shaft. Then they are carried forward to the front edge of the hopper, where teeth attached to the sides of the frame split the chunks as the ribbon of the screw forces the chunks against the teeth.

The shaft of the screw is 6 in. in diameter. Two ribbons of boiler plate are welded to the shaft. The large ribbon is of 1¼-in. plate and bent to make a diameter of 30 in. The small ribbon is 18 in. in diameter and made of ¾-in. plate. The ribbon extends along 4 ft. of the shaft. The screw turns at 15 r.p.m., is driven by a 30-h.p. 1,200-r.p.m. motor equipped with a 20-in. fly wheel, and is direct connected to a 325:1 reducing gear which is connected by a sprocket and chain drive to the shaft. Originally only the larger ribbon was used and the first crusher cost about $3,000 to build. Maintenance costs have been negligible. The crusher, including hopper, drive mechanism and supporting structure for travelling over the conveyor belt, weighs 22,000 lb.

Mobile drum factory

A mobile factory consisting of a number of lorries which, when grouped together, form a complete unit for the production of steel drums, was recently demonstrated in London. The mobile factory has been built by the van Leer Co. in Holland for the manufacture of drums at point of use in those cases where a static unit is not justified and where the transport of finished drums would prove too difficult or costly. The lorries form a production line of 110 ft. manned by ten operatives. The main operations are shearing of steel sheets, rolling of cylinders, spot welding, side-seam welding, expanding of rolling hoops, flanging, corrugating, seaming preformed and painted tops and bottoms, testing by compressed air, spray painting and paint drying by infra-red. Power is supplied by a 200-kVA diesel generator. At the demonstration a completely finished 10-gal. oil drum was produced within 29 min. of the arrival of the first vehicle in the demonstration hall.

Continuous soap cutter

A new machine cuts an extruded bar into pieces of predetermined length and weight with, it is claimed, absolute accuracy. It does this with a minimum use of power, being driven by the bar itself as it emerges from the extruding machine. The rate of cutting is automatically controlled by the speed of extrusion and the cut pieces cannot vary in weight and size. In this gentle action there are no wires to break and no fast-moving parts to wear out. The product is cut by a series of blades which are mounted on brackets linked together to form an endless chain. The chain travels round two guide wheels and over a continuous track, being driven by the pressure of the extruded bar against the blades.

The bar travels along a small belt conveyor under the blades, which gradually penetrate it and cut it cleanly into pieces.

There are no scrap pieces and the product is not bruised while being cut. A second conveyor then removes the cut pieces. A $\frac{1}{4}$-h.p. geared electric motor is fitted to drive this conveyor. The motor serves also to overcome the slight frictional resistance of the cutter chain. The chain runs on ball-races and the conveyor below the blades is supported on a closely-spaced roller track; friction is thereby reduced to a minimum.

The machine, type C.9, is now being produced by Henry Simon Ltd.

Fire-extinguishing system

A U.S. firm, Walter Kidde, has developed a built-in fire-extinguishing system for use in unfrequented areas of industrial plants. The supply source of the multi-space fire-guard system is made up of storage cylinders manifolded together for simultaneous discharge. Cylinder banks can be arranged in groups. The carbon dioxide discharge pipeline from the cylinder bank is connected to a direction valve frame, where there is a valve for each area to be protected. When the fire detector receives a fire signal, it operates a directional valve controlling a group of cylinders, opens the cylinder heads, and the carbon dioxide under its own power is carried through the discharge piping and emerges as an inert gas from the nozzles. The gas expands to 450 times its stored volume, and smothers the fire by excluding oxygen.

Non-lubricated plug valve

A new non-lubricated plug valve for operation in normal to below-zero temperatures on all types of gaseous and volatile liquid service has been announced in the U.S.A. The non-lubricating feature of the valve eliminates daily maintenance and free rotation. Elimination of wear, scoring, galling or sticking is said to be ensured by the retraction of the valve seats before the rotation of the plug. In the closed position the individual seats expand against body ports, and seat inserts can be easily replaced without disassembling. The valves are available in screwed or flanged end, in sizes from 2 to 8 in. for cold working pressure from 230 to 2,000 p.s.i., while sizes of 2 to 4 in. are available for cold working pressures of 3,000 and 5,000 p.s.i. The bodies and covers are of cast steel, the seats of stainless steel, monel bronze, or *Hycar*-bonded stainless, and special materials can be supplied for specific requirements.

New Plant and Equipment

Fill in the Enquiry Coupon on page 138 for further details of the equipment and plant described in INTERNATIONAL CHEMICAL ENGINEERING.

Meetings

Institution of Chemical Engineers

March 9. 'Liquid-Liquid Extraction.' Introduction by A. S. White. Part 1, 'Flooding Rates in Packed Columns,' by F. R. Dell and H. R. C. Pratt. Part 2, 'Hold-up/Pressure Drop in Packed Columns,' by R. Gayler and H. R. C. Pratt. Part 3, 'A Study of Droplet Behaviour in Packed Columns,' by J. B. Lewis and H. R. C. Pratt,' 6.30 p.m., Burlington House, Piccadilly, London, W.1.

March 10. 'The Reactivation of Bone Charcoal,' by W. L. Howe and A. M. Moult, 2.30 p.m., the University, Liverpool.

March 15. Graduates' and Students' Section. 'Chemical Engineering and our Institution,' by L. L. Gush, 7 p.m., City Library, Chester.

March 16. Graduates' and Students' Section. 'Chocolate Manufacture in Scandinavia,' by M. Coleman, 6.30 p.m., Caxton Hall, London, S.W.1.

April 5. Symposium on 'Packed Columns': 'The Performance of Packed Absorption and Distillation Columns with Reference to Wetting,' by H. R. C. Pratt; 'Features in the Design of Packed Towers,' by G. J. Williamson; 'Performance of Carbon Grid Packing,' by W. S. Norman; 'The Operation of Commercial and Semi-Commercial Steadman Packed Columns,' by F. Morton, 11 a.m. and 2.30 p.m., the University, Birmingham.

April 6. 'The Recovery of Bromine from Sea Water,' by R. O. Gibson, 6.30 p.m., Reynolds Hall, College of Technology, Manchester. Joint meeting with Chemical Engineering Group of Society of Chemical Industry.

April 11-14. Graduates' and Students' Section. Convention and annual general meeting, London.

Society of Chemical Industry

March 13. Chemical Engineering Group. 'Possible Applications of Gas Turbines in the Chemical Industry,' by I. Lubbock, 5.30 p.m., Burlington House, Piccadilly, London, W.1.

March 15. Road and Building Materials Group. 'Fibre Building Boards, their Manufacture, Properties and Uses,' by H. Rose, 6 p.m., Institution of Structural Engineers, 11 Upper Belgrave Street, London, S.W.1.

March 20. Agriculture Group. 'Agricultural By-Products and their Industrial Utilisation,' by F. N. Woodward, Institute of Seaweed Research, 2.30 p.m., Royal College of Science, London, S.W.7.

March 28. Chemical Engineering Group. 'Relation of the Chemical Engineer to the Chemist and Production Engineer in Industry,' by A. H. Loveless, the University, Birmingham.

April 5. London Section. 'Recent Trends in Fuel Research,' by A. C. Monkhouse, 7.30 p.m., Kings Fare Restaurant, King's Street, Luton.

Chemical Society

March 10. Discussion. 'Training of Scientists,' 2.15 p.m., Washington Singer Laboratories, Prince of Wales Road, Exeter. Joint meeting with Royal Institute of Chemistry and Society of Chemical Industry.

March 14. 'Production and Applications of Radioactive Isotopes,' S. H. Bales, 7.45 p.m., University College, Upper Merrion Street, Dublin.

March 15. 'Materials of Construction for Chemical Plants,' by N. P. Inglis, 7.30 p.m., North British Station Hotel, Edinburgh. Joint meeting with Royal Institute of Chemistry and Society of Chemical Industry.

March 15. 'Recent Progress in the Study of Metallic Oxidation and Corrosion,' by W. J. H. Vernon, 7 p.m., the University, Bristol. Joint meeting with Royal Institute of Chemistry and the Society of Chemical Industry.

March 20-21. Anniversary meeting. Presidential Address, 'Concepts in Catalysis, the Contributions of Paul Sabatier and Max Bodenstein,' by Prof. Rideal, 4 p.m.; reception, 6.30-8.30 p.m.; Burlington House, London, W.1. *March* 21. Annual general meeting, 11.30 a.m., Burlington House; visits to research establishments, 12.15 p.m.; anniversary dinner, 7 p.m.

Institute of Fuel

March 9. 'The Down-Jet Coke Burner,' by F. B. Karthauser, F. F. Ross and G. C. H. Sharpe, 6 p.m., Central Library, Alexandra Road, Swansea.

March 9. 'Drying of Animal By-Products,' and other papers, 6 p.m., North British Hotel, Edinburgh.

March 14. 'Drying in the Textile, Paper and Allied Industries,' by R. R. Clegg, and other papers, 10.30 a.m. and 2 p.m., Bradford.

March 15. 'Drying of Pastes, Powders and Crystals,' by L. Clegg and S. C. Jackson; 'Drying of Liquids, Solutions and Slurries,' by B. N. Reavell; 'Drying of Milk,' by S. A. Wilcox, 5.30 p.m., Institution of Mechanical Engineers, London, S.W.1.

March 21. 'Drying in Agriculture,' by W. H. Cashmore, and other papers, 10 a.m. and 2.30 p.m., the Technical College, Lincoln.

April 3. 'Underground Gasification of Coal,' by C. A. Masterman, 5.30 p.m., Institution of Mechanical Engineers, London, S.W.1.

April 12. Above paper repeated at 6.15 p.m., Gas Showrooms, Nottingham.

Institute of Metals

March 15. 'Wetting of Metal Surfaces,' by J. Bailey, 7 p.m., 4 Grosvenor Gardens, London, S.W.1.

March 22. 'Adhesives for Metals,' by C. J. Moss, 6.30 p.m., James Watt Memorial Institute, Great Charles Street, Birmingham.

Achievements and Aims of the Indian Chemical Industry

INDIA'S progress in developing her own basic chemical industries and her plans for the future were reviewed recently by the president of the Indian Chemical Manufacturers' Association, Dr. K. A. Hamied. He was speaking at the 11th annual meeting of the association in New Delhi, in the presence of the Prime Minister, Mr. Nehru, who inaugurated the proceedings.

Platform at the annual meeting of the Indian Chemical Manufacturers' Association. *Left to right:* Mr. Nehru, Prime Minister; Dr. K. A. Hamied (President of the Association); Dr. L. A. Bhatt (Incoming President); and Mr. Ganpathy (Secretary).

Heavy chemicals

In the field of heavy chemicals, Dr. Hamied claimed substantial progress. Sulphuric acid output had reached 150,000 tons p.a., compared with the present home demand of 100,000 tons. Because of the sulphur shortage, development of production of the acid from calcium sulphate was suggested. Soda ash requirements are not yet met from Indian plants, which produce only 54,000 tons p.a. against an annual requirement of 90,000 tons. More soda ash plants are needed, therefore. Supplies of home-produced caustic soda are even smaller, only 18,000 tons to meet a demand of 50,000 tons. However, when plants now being constructed are finished, India should be self-sufficient. From the present output of caustic soda, some 6,500 tons p.a. of chlorine could be produced, but there is as yet not enough demand to take even this quantity, with the result that chlorine is run to waste and caustic is dearer than it should be. Development of chlorinated products is recommended, therefore. Calcium carbide is an important chemical not yet produced in India, but arrangements are being made to build a plant for its manufacture as soon as cheap electricity is available from the Damodar Valley project, presumably a hydroelectric scheme.

One of the most publicised Indian chemical projects is the giant Sindri plant, designed to produce 350,000 tons of ammonium sulphate annually. In spite of nearly four years' work, this plant is not yet in production, and even when it is Dr. Hamied believes there will be a large gap between production and demand. Some 700,000 tons p.a. of superphosphates are also needed, but there is no indication that the necessary plants are being built.

Drugs and fine chemicals

Discussing the pharmaceutical industry, Dr. Hamied claimed that it was now producing enough alkaloids, sera, vaccines and other standard pharmacopoeial preparations to cover the home needs and even to export. Fine chemicals based on ethyl alcohol, produced in abundance in India, were also in ample supply from domestic sources. The great deficiency in the drug field, which almost outweighs present advantages, is the lack of coal-tar intermediates on which the manufacture of chemotherapeutics like sulphonamides and anti-malarials depends. Until an Indian coal-tar chemical industry has been established successfully, dependence upon foreign producers for these raw materials will continue. Dr. Hamied did not mention the other major class of drugs, the antibiotics, a curious omission in view of India's deficiencies in antibiotic production facilities.

In spite of the progress of India's pharmaceutical industry, it seems that its products are far from being accepted with the same confidence as the well-known branded preparations of foreign manufacture. Dr. Hamied rejected the suggestion that Indian products were inferior and stated that the widespread impression that this was the case was due to propaganda by foreign interests. Since the final criteria of a product are its price and quality, it seems that the prejudice against Indian products is not due solely to 'foreign propaganda.'

To encourage development of the pharmaceutical industry Dr. Hamied demanded import barriers on the lines adopted by Western countries. He criticised the fact that liberal imports of finished drugs and medicines were permitted, but did not say how India's needs are to be met if they are stopped. On the other hand, he demanded duty-free and uncontrolled imports of pharmaceutical raw materials, plant and equipment.

Automatic control in oil refineries

The successful operation of modern large oil refineries is directly attributable to the use of automatic control instruments. Fractionation forms the basis of practically all oil-refining processes; temperature, pressure, flow and liquid level are the commonest factors necessitating control. All are controlled in essence by the flow of particular substances, the rate of flow being controlled by means of a throttling valve actuated by compressed air acting on a diaphragm.

Methods of control of temperature and pressure have advanced in recent years. The application of electronics to temperature control has resulted in the thermocouple current being amplified as much as $2\frac{1}{2}$ million times. This has resulted in the building of an instrument which will record as many as 40 temperatures in 1 min. The electronic potentiometer with its continuous balance system detects the change in temperature and makes a suitable correction in the control-valve setting before the galvanometer potentiometer has recognised the fact that any change in the temperature has occurred. Such response permits the use of a narrower throttling range with consequent greater hold on the process.

The use of super-fractionators for the separation of narrow boiling hydrocarbons has necessitated the use of a vapour-pressure controller, that is, one correcting for changes in temperature and pressure. Its commonest use is as a composition controller in the separation of binary mixtures. It consists of a temperature bulb, partly filled with one of the materials being fractionated, and a pressure connection, both being located at the same point in the column. By holding a zero differential pressure on the vapour-pressure controller the composition on the tray will remain constant irrespective of changes in temperature and/or pressure.

The fluid catalytic process presents a good example of the use of automatic control instruments, as it has a total of 88 controllers and 54 recorders.—C. B. McLaren, *Royal Aust. Chem. Inst. J.*, 1950, 17 (7), p. 259.

World News

GREAT BRITAIN

Zirconium metal made

The production of zirconium metal on a commercial scale has recently been started by Murex Ltd. Previously only very limited supplies were available in England and even now full production has not yet been attained. Sheets down to .005 in. in thickness, 6 to 7 in. in width and 2 to 3 ft. in length are now being made, and it is expected that sheets up to 12 to 15 in. in width will be available in the course of the next few months. Rods down to 2-mm. diam. can be supplied and drawn zirconium wire may also shortly be available.

Zirconium is claimed to have excellent resistance to corrosion. Hydrochloric acid and fused alkalis have practically no attack on this metal, while its resistance to attack by nitric, phosphoric and sulphuric acids is only slightly less than in the case of tantalum. This property renders it very suitable for use in chemical plant where corrosion resistance is required.

Record output of fertiliser

Production and consumption of sulphate of ammonia in the U.K. has been increasing steadily during the past few years to reach a record value last year, according to the annual report for 1949-50 of the British Sulphate of Ammonia Federation Ltd. Actual production of the fertiliser (nitrogen content varying from 20.4 to 21.1%) amounted to 924,500 tons in 1949, compared with 901,000 tons in 1948, 796,700 tons in 1947 and 483,300 tons in 1939. Home agricultural consumption has increased considerably to a total of 748,115 tons for the fertiliser year 1949-50, compared with 179,700 tons for 1938-39. Although production has doubled over the past ten years, domestic consumption has increased four-fold.

Exports of sulphate of ammonia from the U.K. were 15% greater during 1949-50 than in 1948-49, although they were by no means a record.

The research department report steady progress in the production of salt of improved quality and, during the year, 36% of the total by-product salt was of grade 1 standard.

Export restrictions on chemicals

Under an order made by the Board of Trade, which came into operation on February 12, export licences are required for all destinations for candles and certain similar articles, certain fatty acids, antimony, vanadium and zinc and their alloys in certain forms, nickel ores and concentrates, some further types of used steel materials, feathers and down, cobalt compounds, naphthalene, sulphuric acid and anthraquinone.

From that date also licences are required for corundum, certain plastic materials, carbonyl iron powder, molybdenum carbides, certain scientific apparatus and various chemicals, for export to all destinations other than those specified in Part II of the Third Schedule.

Certain vegetable oils were exempt from export licensing control from February 12.

North-western chemical engineers meet

The sixth annual meeting of the north-western branch of the Institution of Chemical Engineers was held recently in Manchester. The chairman, Mr. E. J. Dunstan, said that the branch was progressing well and attendance at meetings was excellent. Because of the poor response to enquiries, it was decided not to form a branch graduates' and students' section; the matter was still being studied, however.

After the meeting, Mr. Oliver Lyle talked about 'Sugar as the Basis of Life,' and traced the origin of sugar through to its commercial manufacture, touching on its chemical engineering significance.

Among the guests at the annual dinner following the meeting were Prof. Garner, director of the chemical engineering department of Birmingham University, Dr. E. T. Hoblyn, director of the British Chemical Plant Manufacturers' Association, Dr. J. B. Brennan, general secretary of the Institution of Chemical Engineers, Dr. F. A. Freeth, I.C.I., and Mr. Lyle.

New officers of the branch are: G. Brearley, *chairman;* E. J. Dunstan, *vice-chairman;* J. S. Hunter, *hon. secretary;* and F. H. Bramwell, *hon. treasurer.*

Mond Nickel fellowships

The Mond Nickel Fellowships Committee have awarded five fellowships for 1950 for the study of metallurgical practices in a number of countries, including the U.K., U.S.A. and Canada.

At a later date applications will be invited for awards for 1951. Full particulars can be obtained from the secretary, Mond Nickel Fellowships Committee, 4 Grosvenor Gardens, London, S.W.1.

Institute of Metals awards

Dr. R. W. Diamond, Consolidated Mining & Smelting Co. of Canada Ltd., has been awarded the platinum medal of the Institute of Metals in recognition of his work in connection with researches on differential flotation as applied to the complex Sullivan ore, and as manager of the largest combined copper and lead producer in the world.

Mr. Christopher Smith, James Booth & Co. Ltd., Birmingham, received the W. H. A. Robertson medal, and Prof. G. V. Raynor, University of Birmingham, the Walter Rosenhain medal.

Price increases

Soap. The sharp rise in costs of raw materials used in making soap has resulted in increased maximum retail prices for soap and soap products being approved by the Ministry of Food. Hard soap and soap flakes have gone up by 1d./lb., while toilet soap now costs ½d. more per 3-oz. tablet. The new prices became effective on February 4.

Oils and fats. The prices of unrefined oils and fats and technical animal fats allocated to primary wholesalers and large trade users were increased last month by amounts varying between £9 and £20/ton.

Phthalates. Owing to further increases in raw materials and other costs, British Industrial Solvents Ltd. have raised the price of all phthalates. The new prices range from 1s. 9¼d./lb. for dimethyl phthalate to 3s. for dioctyl phthalate.

Zinc oxide output

From January to November last year, 50,400 tons of zinc oxide were produced from 26,900 tons of virgin zinc and 17,400 tons of scrap. This represented 12% of the consumption of the former and 20% of that of the latter. Allocation of zinc for oxide manufacture in February was only 70% of average monthly consumption during the first nine months of 1951.

Top prices for non-ferrous scrap

A new order by the Ministry of Supply fixes the prices of non-ferrous scrap. This is to discourage hoarding and stabilise scrap prices so that they are in reasonable relation to the prices of the virgin metal. Recently scrap metal has been offered at prices above those of virgin metal.

Gas Development Committee

The Industrial Gas Development Committee, which was formerly a committee of the British Gas Council, will continue its work as a committee of the Gas Council.

Mr. H. R. Hems, Industrial Gas Officer of the West Midlands Gas Board, and Mr. R. F. Hayman, of the North Thames Gas Board, have been elected chairman and deputy-chairman, respectively, for the ensuing year.

New B.I.S.R.A. deputy director

Mr. W. C. F. Hessenberg has been appointed deputy director of the British Iron and Steel Research Association, of which Sir Charles Goodeve is director. Mr. Hessenberg has been head of the Association's Mechanical Working Division since March 1947, and retains this position.

British Council courses

The British Council's 1951 programme of short courses and summer schools in Great Britain for overseas visitors includes: 'Water Pollution,' London and Birmingham, May 21 to June 2; 'Insecticides and Weedkillers,' Rothamsted and Oxford, June 18 to July 3; and 'Industrial Stan-

dardisation as Developed in the U.K.', London, August 27 to September 8.

Thermostatically-controlled metal-testing laboratory

In order to concentrate more attention on the creep properties of metals, a large thermostatically-controlled room to house all the creep- and fatigue-testing machines has been set up by the British Thomson Houston Co. Ltd., Rugby. Steels that are specially designed for creep resistance are 'precipitation-hardened' by carbides and intermetallic compounds and, by means of creep experiments performed at different stresses, work on the theoretical relationship between creep strain and stress for such materials is being carried out.

Blading and bolting materials for gas and steam turbines are tested at stress and temperature conditions under which the components will operate. The relaxation property of molybdenum-vanadium steel has been determined by simulating the daily stress cycle resulting from the cooling and reheating of the turbine.

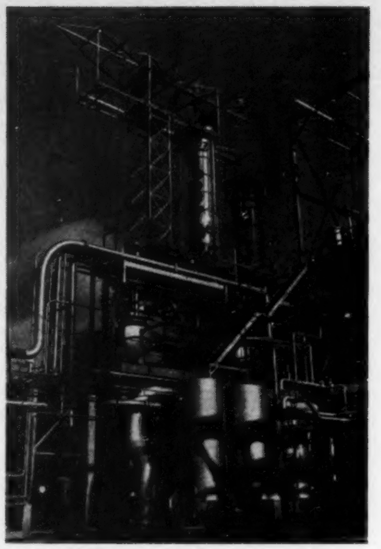
An apparatus has been constructed for examining high-temperature steam corrosion of metals. Good corrosion resistance must be complementary to creep resistance in steam turbines operating at higher temperatures, and it is desirable that the scale formed should adhere to the metal and thus help to protect it from further oxidation. Small cylindrical specimens are subjected to heating and cooling in an atmosphere of nitrogen and, while at high temperature, a steady stream of superheated steam passes over the metallic surfaces. At the conclusion of the test, measurement is made of the extent of corrosion and the degree of adherence of the oxide scale.

Building research papers

The national building studies of the D.S.I.R. are divided into three series—bulletins, special reports and technical papers. There has been some indication that the technical papers are being purchased under the misapprehension that they deal with matters of immediate and direct interest to the builder. They are not intended for the building industry in general, for most of the material they contain is of interest only to those engaged on research and development work. To emphasise the different nature of the technical papers, the series is now being renamed 'research papers.'

New comptroller of Metrovick

Following the retirement on December 31 of Mr. J. G. Lowe, comptroller and secretary of Metropolitan-Vickers Electrical Co. Ltd., Mr. E. Salmon has been appointed comptroller and Mr. D. Thomson secretary as from January 1, 1951. Mr. W. D. Taylor has been appointed to succeed Mr. Lowe as secretary of Metropolitan-Vickers Electrical Export Co. Ltd. as from the same date.



NEW REFINERY UNIT

The new plant at the Shell Haven, Essex, refinery came into operation recently six weeks ahead of schedule and is now manufacturing petrol and other oil products at a rate of 1,450,000 tons p.a. Production is at the moment confined to one distillation unit and ancillary facilities. The full refinery, scheduled for completion by 1952, will have a capacity of 2,000,000 tons p.a., which, together with that from the Stanlow refinery and the 1,500,000 tons p.a. installation at Heysham, Lancs, will make Shell's total 6,500,000 tons p.a. by 1952, the largest of any refinery operator in the U.K. The photograph shows columns C_4 and C_5 of the plant.

DENMARK

Cellulose factory project

A factory for the production of cellulose is to be erected in Denmark. Details have not been completed, but the factory is expected to produce some 7,000 tons of cellulose p.a., which will be sold in Denmark at around Kr.7,500 per ton, half the price currently paid for imports. The cost of building the factory is expected to be around Kr.6,000,000. Annual cellulose imports now amount to some 100,000 tons.

AUSTRIA

More rayon pulp

The Lenzinger Zellulose und Papierfabrik A.G. supplied 19,180 tons of rayon pulp to the rayon works Zellwolle Lenzing A.G. in 1950 and plan to step up deliveries to 22,000 tons this year and 26,000 tons in 1952. In addition, the pulp works have agreed to obtain in 1951 about 5,000 tons and, in 1952, approximately 6,000 tons of paper pulp from other sources, and in exchange to produce the same quantities of rayon pulp for the rayon works.

New nickel sulphate plant

Experimental production has started at a new nickel sulphate plant built by the State-owned Montanwerke at Brixlegg. When the full production of 300 tons p.a. is reached, it will make an important contribution to Austria's foreign exchange balance, not only by eliminating imports of this material, on which Austria has had to rely hitherto, but also by annual exports of approximately 150 to 180 tons of nickel sulphate. The plant cost 3,000,000 Austrian schillings, of which 1,800,000 were made available from E.R.P. funds.

GREENLAND

Nickel project abandoned

Nickel found in Greenland last year is not to be exploited. Danish experts stated that the yield would be around 1 kg./ton of ore, much less than was required to make full-scale exploitation worth while.

In recent years, deposits of gold, silver, copper and lead have been found in Greenland. It is hoped that the lead deposits may prove large enough to justify exploitation. (See INTERNATIONAL CHEMICAL ENGINEERING, January 1950, p. 43.)

SPAIN

Plans to produce more lead

Average lead-ore extraction in Spain during 1950 is estimated at 3,725 tons/month, against a monthly average of 3,670 in 1949 and 3,473 in 1948. Production of lead is estimated to have averaged 2,748 tons/month during 1950, against 2,280 in 1949 and 1,838 in 1948.

Efforts are at present being made to find and exploit new sources of lead in Spain, not only for the domestic but also the export market. In the province of Jaen, near Linares, soundings are being carried out to ascertain the extent of present ore-bearing zones, and the construction of a 6.5-km.-long drain tunnel is planned.

A washing installation for the daily treatment of 100 tons of lead and zinc ore, which will yield 700 tons of lead concentrates (70%), has been constructed at the Florida mine in Santander, which began working in 1949. The Manto de Azules mine also began working in 1949, and a washing installation is being built there (near La Union, in the province of Murcia) for the treatment of 300 tons/day of lead and zinc ore with an annual production of 2,400 tons of lead concentrates. A further washer is being installed in Mazarron (Murcia) for the treatment of 200 tons of lead ore, and a yearly production of 1,800 tons of concentrates. In the Sierra Almagrera mines, drain tunnels are being pierced to permit working the mines below the present level of the waters. In the province of Granada, a washing installation is to be built in the village of Tablones for the treatment of 150 tons/day of ore and a yearly production of 2,000 tons of concentrates. New ovens for the treatment of 20 tons/day of ore are to be built at Renteria, near San Sebastian, in north Spain. At Villaler in the province of Lerida, a factory is being built with a capacity of 4,800 tons p.a. of lead metal.

FRANCE

Fertiliser and tar products from Saar steel industry

A total of 255,000 metric tons of basic slag was produced by the Saar iron and steel industry last year, compared with 273,000 tons in the preceding year. As the output depends on the production of mild steel, the higher 1949 figure is explained by the existence of a carry-over from the preceding year.

The principal customer last year was France, who purchased 186,000 tons, followed by Germany with 14,000 tons. A further 9,000 tons went to other foreign countries, while 25,000 tons were bought by Saar customers.

The industry also produced last year 28,600 tons of ammonium sulphate (against 28,800 in 1949), with France and the Saar the only customers; 166,000 tons of raw tar (against 169,000 in 1949), of which 59,000 tons went to France and 1,000 tons to other foreign consumers; and 51,600 tons of crude benzole (against 52,800 in 1949), most of which was used within the Saar.

GERMANY

New sulphur plant

A sulphur plant at the Hansa mines of Gelsenkirchener Bergwerks A.G., Dortmund, has been completed. Production at the rate of 5 metric tons daily has started.

Paint and lacquer industry

The paint and lacquer industry of Western Germany centres in the Rhineland-Westphalia area, which has a capacity for about 50% of total production. Hamburg is the second producing area, accounting for about 15% of the output. Bavaria, Hesse and Württemberg-Baden produce about 10% each, and the remaining 5% comes from Schleswig-Holstein and Niedersachen. Berlin and Saxony were important pre-war paint centres. Leipzig, Dresden and Halle each have large lacquer plants, but the position of the industry in the Soviet zone is said to be unfavourable.

Hamburg and vicinity has six large paint factories and 22 smaller ones. In addition, several plants make specialities such as furniture polishes and stains, alcohol and cellulose lacquers, and ship's-bottom paints. All large Hamburg plants have modern equipment.

YUGOSLAVIA

Progress in copper production

The Bor mine in Yugoslavia, Europe's largest individual source of copper, produced 40,000 tons of blister copper last year. It was feared at one time that the Bor deposits would be exhausted in a few years, but recent discoveries have proved the opposite.

A new mine at Majdanpek in eastern Serbia is claimed to be the second largest in Europe and, during 1950, intensive surveying and research were carried out. There are also substantial deposits of iron ores in the Majdanpek region.

Two-thirds of Yugoslavia's copper output have hitherto been exported in the raw state, while the remaining third has been processed in the Bor electrolytic plant. The large electrolytic plant now being erected at Bor will be able to process the whole of the copper output; half the work on the plant is scheduled to be completed this year.

Provision is also being made for further processing. A modern copper-rolling mill in course of erection at Sevojno is due to be completed in 1952. It is being built by the Yugoslav Youth Organisation (Omladina Jugoslavije) and should be able to turn out some 21,000 tons p.a. of finished products.

ITALY

New resin factory

A new resin factory connected with the American Reichhold Chemical Group has been opened near Naples. There is understood to be a similar factory in Milan which has been producing resin for varnish since 1942.

Magnesite from the sea

A large plant, similar to those existing in the U.K., is to be established to extract magnesite from sea water and provide the Italian refractories industry with an essential raw material.

The Italian refractory industry is reported to consist of about 65 firms, employing 12,000 workers and producing about 300,000 tons p.a.

Methane production up

Production of methane is reported to be developing rapidly. According to the vice-president of A.G.I.P., the company's output is now about 1,400,000 cu.m./day, equivalent to some 2,100 tons of coal. Within a year, deliveries to industries are expected to reach 2,000,000 cu.m./day, equal to 3,000 tons of coal.

NORWAY

New plant may double aluminium output

Norway is to build a new plant to manufacture aluminium at the rate of 50,000 tons p.a. Hr. Drogseth, of the Norwegian Ministry of Industry, confirms that negotiations for building this plant have made good progress: 'If the necessary decisions are taken in the next couple of months, it should be possible to start construction this spring and production in 1954.' The plant will be built at Sunnadalsora in west Norway, and power will be supplied by the Aura hydroelectric station. Production at first would be at the rate of 40,000 tons p.a., but could later be increased to 50,000 tons. Production at this rate would double Norway's total aluminium production, which last year amounted to 45,000 tons. The new plant will be one of the biggest investment projects since the war. It will cost £10,000,000 to build and money will also have to be spent on increasing the capacity of the Aura hydroelectric station. At present prices, 40,000 tons of aluminium can be sold for £5,500,000, and net currency earnings would amount to over £3,000,000. The plant will employ up to 900 men when in production.

Nitrogen output in 1950

Norway's largest industrial concern, Norsk Hydro, last year produced 146,000 tons of pure nitrogen, compared with 104,000 tons the previous year. Capacity is now 160,000 tons p.a., equivalent to over 1,000,000 tons of nitrates. The value of Norsk Hydro's sales last year was £10,650,000, compared with £7,900,000 the previous year. A net profit of £625,000 was made. Norsk Hydro's assets, according to the annual report, were put last year at £24,000,000.

SWEDEN

Chlorine and alkali plant

A new Kr.1,800,000 plant to produce 3,000 tons of chlorine and 4,000 tons of alkali p.a. is to be erected at Bomhult, in the north of Sweden, by Korsnasverken, one of the country's most important pulp producers. At present the chlorine and alkali have to be imported.

New sulphur-reduction process

A method of reducing the sulphur content of iron has been used on an industrial scale by the Domnarvet Iron Mills in central Sweden for the past six months.

It is stated to have yielded surprisingly favourable results. The method, which is known as 'Kalling's roller-furnace method' after its inventor, Prof. B. O. Kalling, involves the use of coke, but the final product is claimed to be equal in quality to Swedish charcoal pig-iron.

Under the Kalling process the pig-iron flows from the blast furnace into a basin which has a capacity of 12 tons. From there it is tapped into a roller furnace which is being driven at the rate of 34 r.p.m. Burnt lime in the proportion of 2% of the total weight of the pig-iron, as well as some coal dust, are added, and the furnace is then closed and made to rotate. The percentage of sulphur decreases to the desired level within 30 min. When tapped into the roller furnace, the pig-iron contains 0.09% of sulphur. When the furnace starts to rotate the percentage of sulphur decreases, first very quickly and then more slowly to a final value of only 0.005%. At the same time the proportion of silicon is reduced to about 10%.

The sulphur is absorbed by the burnt lime, producing calcium sulphide; the oxygen which is then freed unites with the silicon, yielding silicon-dioxide.

SOUTH AFRICA

Oil-from-coal plant

The Union's first oil-from-coal plant will be built near Coalbrook in northern Orange Free State, according to a statement by the S. African Coal Oil & Gas Corp. Coalbrook is situated on the banks of the Vaal river and is the major source of coal in S. Africa. It is believed that 15 to 20% of the Union's petrol needs will be met by the plant.

Tung oil factory

According to the managing director of the East Asiatic Co. (S.A.) Ltd., a factory is being erected on the company's plantation in Nyasaland for expressing tung oil. It is hoped shortly to complete the cultivation and planting of the 4,000-acre estate, which will then be the largest single area in Nyasaland under tung.

SOUTHERN RHODESIA

Gold refinery planned

The establishment of a gold refinery is under discussion by the Joint Standing Committee of the Chamber of Mines of Rhodesia and the Rhodesia Mining Federation, according to the *Natal Mercury*. Rhodesian mines were said to be paying more than £80,000 p.a. in refining charges and assay fees. It was suggested that the establishment of a Rhodesian refinery would not only keep money in the country, but would reduce charges to the S. Rhodesian mines. The idea was that capital should be raised locally by private enterprise. The refinery would not be Government-controlled.

BELGIAN CONGO

Plastics and soap industries expand

Several small industries, including the manufacture of plastics and soap, are expanding in the Belgian Congo. Activity in the construction industry may lead to the establishment of larger enterprises such as paint factories.

GOLD COAST

New aluminium project

The establishment of an aluminium factory is proposed in connection with the hydroelectric development of the Volta River. A preliminary report by Sir William Halcrow & Partners, consulting engineers, has been accepted by the Gold Coast Government. The scheme involves the construction of one of Africa's greatest dams and an 800,000-kW. hydroelectric plant. The project, at Adjena, about 65 miles from Accra, would provide electricity to produce aluminium from bauxite located at Yenanin in Ashanti Province.

About £100,000,000 will be required to finance the complete scheme, which includes the construction of railway lines, and harbour facilities as well as an irrigation system.

EGYPT

Cement output up

Production of cement totalled 963,000 tons in 1950, against 876,000 the year before, and 386,000 tons in 1939, according to the Ministry of Supply. A further increase in output during the current year is predicted as a result of scheduled imports of new machinery and equipment.

Growing demand for fertilisers

Present consumption of fertilisers in Egypt is given as 700,000 tons p.a. This figure increases annually by 10 to 20% owing to impoverishment of the soil, and a meeting was recently held by chemical fertiliser importers in conjunction with the Ministry of Agriculture to study ways and means of assuring the country's present and future requirements of fertilisers.

Current domestic production consists of 60,000 tons of superphosphates annually. Imports for the current season should amount to 700,000 tons of fertilisers, including nitrate of lime, sulphate of ammonia and sodium nitrates. To cover her requirements up to the end of the 1951-52 season and to build up stocks, it is estimated that Egypt must import 1,400,000 tons.

ISRAEL

New factories

An edible oil refinery and extraction plant has been set up at the oil and soap factory near Petah Tikva.

An electrode plant said to have a capacity of 2,500 tons p.a. has been opened near Haifa. Two-thirds of the production is reported to be for export.

Cement production

An average of 37,000 tons/month of cement is now produced at the Nesher works, Haifa, compared with 20,000 tons/month in 1949.

Plastics industry expanding

The Israel Plastic Manufacturers' Association now numbers 16 member factories and more plants are being erected. The 16 firms, most of whom work double shifts, have a joint annual turnover of about £I1,000,000 and produce over 1,000 different lines. However, in view of the inadequate utilisation of capacity, the plants are unable to meet all local requirements of plastic goods.

Raw materials have to be obtained from abroad and, due to the country's shortage of foreign exchange, import permits have been sparse over the past year. Manufacturers have therefore resorted to barter agreements, supplying Israel-made textiles against plastic moulding powders. They are now trying to increase this form of supply, as additional quantities will be required for use with $100,000 worth of new machinery purchased with the U.S. loan to Israel.

PAKISTAN

Plans to produce eucalyptus oil and tannic acid

Industrial research laboratories in the Punjab are experimenting on the extraction of oil from eucalyptus trees found in the Muree hills and other parts of Pakistan. If the oil extracted is found to contain the medicinal properties which are present in, for instance, Australian eucalyptus oil, extraction will be undertaken on a large scale.

'Pilchi,' a tree found on the banks of the Ravi river, has been found to contain tannic acid in sufficient quantity to supply Pakistan's ink industry.

Rawalpindi grass suitable for paper manufacture

Experiments on a number of types of grasses which grow in the dry hills of the Rawalpindi district have shown that a large-scale paper factory could be set up in the Punjab, which could be supplied easily with local raw materials. The rice straw which is produced in large quantities all over the Punjab and which usually goes to waste, has also been found to be a good raw material for making cardboard.

Fertiliser plant planned

A fertiliser plant with a capacity of between 50,000 and 80,000 tons p.a. is being planned by the Pakistan Government and will probably be located in the Mari Indus district of Mianwali.

INDIA

Steel development possibilities surveyed by American expert

India, with her natural resources, could become a major producer of steel in about four years, according to Mr. P. V. Martin, manager of Koppers Inc., Pennsylvania, U.S.A.

He estimated that, if India were to have a new integrated steel plant with an ingot capacity of 1,000,000 tons p.a., in addition to expansion of present Indian steel companies, she would have to spend $250,000,000.

Mr. Martin is assisting the Indian Government in a tariff enquiry on iron and steel.

First penicillin factory

The World Health Organisation and the United Nations Children's Emergency Fund have offered to assist India in setting up her first penicillin factory. The U.N.I.C.E.F. will provide $850,000 worth of apparatus from the U.S. The W.H.O. has offered a grant of $350,000 to pay for the technical experts.

The building of the plant was authorised in December 1949. The project has been undertaken jointly by the Central Government and the Government of Bombay, subscribing 60 and 40% of the capital, respectively.

NEW CHEMICAL HANDLING EQUIPMENT

New equipment for the handling of sulphur and bauxite from steamship to distantly located storage areas has been installed by the American Cyanamid Co. at its Warners plant, Linden, New Jersey. The equipment includes an unloading tower and a new type of boom stacker with auxiliary conveying and distributing equipment and will handle these two materials at the rate of 600 gross tons/hr. The tower crane, a revolving-type crane with large digging bucket, is of a completely new design and features electric-welded structural fabrication. All equipment is run from a remote control station by one man operating control buttons and safeguarded by automatic safety devices. The stacker has a self-contained, projecting lever arm, or boom, supporting a belt conveyor which can be raised, lowered and rotated in a complete circle. It is claimed to be the first completely hydraulic-operated machine of this type.

AUSTRALIA

Plans to raise cement output

We understand from Mr. E. M. Schroder, managing director of the Adelaide Cement Co. Ltd., that the proposed scheme of expansion of his company envisages the installation of a rotary kiln, 11 ft. 3 in. diam. by 440 ft. length, with ancillary equipment. This unit will have a production capacity of 150,000 tons p.a. Erection is due to be completed at the end of 1952. The scheme will cost about £450,000.

The expansion plans of a second Australian company, Metropolitan Portland Cement Co., New South Wales, comprise the installation of new plant at Picton, 50 miles from Sydney, at a cost of £2,000,000. It will have a capacity of 5,000 tons/week of cement (260,000 tons p.a.). Two kilns bought in the U.S. are expected to be operating shortly, while three others from Norway are scheduled to be working within eighteen months.

PHILIPPINES

New steel plant

A new steel plant is to be set up on a 15-acre site near Manila recently acquired by the newly formed Marcelo Steel Corp. The plant will consist of three electric-arc furnaces, an ingot rolling mill and a mill for re-rolling scrap, all housed in a modern structural steel and concrete building to be erected at an estimated cost of 500,000 pesos.

The Philippine Government has granted 6,000,000 pesos to the company, and equipment for the plant has already been ordered. Deliveries are expected to start shortly.

The Government recently stopped the export of all scrap metals in order to conserve supplies for this new plant.

JAPAN

PVC factory

A new company, known as the Japanese Geon Co. Ltd., has been formed to build facilities in Japan for the manufacture of *Geon* polyvinyl chloride, an important plastic.

Principal stockholders are the U.S. firm, B. F. Goodrich Chemical Co., and three Japanese companies, Farukawa Electric Co., Nippon Light Metal Co. and the Yokohama Rubber Co., all of Tokyo.

Industrial salt shortage

The soda industry is reported to be suffering from a salt famine, due mainly to the shortage of sterling and the non-arrival of 160,000 tons of salt which was to have been imported from Communist China. Plans had been made to expand soda ash and caustic soda production capacity this year.

Requirements of salt during the January to March quarter are estimated at 550,000 tons, although imports will only amount to 300,000 tons and present stocks are given as 100,000 tons.

U.S.A.

Carbon black plant to be enlarged

The capacity of the new furnace-black plant under construction near Baytown, Texas, by the J. M. Huber Corp. is to be double that originally planned (see INTERNATIONAL CHEMICAL ENGINEERING, September 1950, p. 428). The first plant will be ready for production in May and the second in November. Total output of HAF-type carbon black will then be 72,000,000 lb. p.a., it is estimated.

More soda ash

Production of soda ash is to be increased by 200,000 tons p.a. by the Allied Chemical & Dye Corp. It is estimated that between eighteen months and two years will be required to complete the scheme. The expansion will be carried out at the Solvay plant at Baton Rouge, Louisiana.

Benzene production plan

Leading U.S. manufacturers propose to increase the output of benzene from petroleum from 12,000,000 to 100,000,000 gal. p.a., if official consent is obtained. It is estimated that the scheme will take two years to complete.

Rubber situation satisfactory

This month (March) all the U.S. synthetic rubber plants should be in operation and, when they reach capacity, will be producing at the rate of 1,000,000 tons p.a. of rubber. Mr. H. S. Firestone, chairman of Firestone Tire & Rubber Co., has forecast that there will be an ample supply of rubber to meet all essential civilian needs and at the same time fulfil all defence needs.

As far as natural rubber supplies are concerned, a National Production Authority representative said that plenty of liquid latex was now arriving from the Far East, but many shipping problems could be eliminated if the latex could be shipped dry. An order allocating latex may soon be issued to bring its use below the present rate of about 6,000 tons/month. Officials are discussing with the industry the reduction of natural rubber content in products where it is technically not necessary. Civilian consumption of natural and synthetic rubber is limited to a total of 83,500 tons/month for January, February and March of this year and is expected to be reduced in the following months.

New process for cast iron

In a new process for producing ductile cast iron, a mechanical mixture of finely divided zirconium-bearing material and finely divided elemental magnesium is added to the molten cast iron. The amounts used leave a residual zirconium content of 0.5% or less and a magnesium content of 0.2% or less. The patent covering this process (U.S. Pat. 2,538,263) has been awarded to the Union Carbide & Carbon Corp.

Paint heat indicator

A new type of paint which varies its colour according to variations in temperature is being used by the Socony Vacuum Oil Co., Inc., U.S.A., in studying the cooling processes of air-cooled internal combustion engines. Colour bars made by different temperatures on an engine coated with this paint are similar in principle to the isobars of a weather map, the company's technicians state. The paint acts as a guide to the correct placing of thermocouples to record internal heat when the engine is in operation. Hitherto, the placing of thermocouples could distort air currents around the test engine sufficiently to affect the accuracy of the recordings.

The formulation of the new paint gives 15 basic shades for original application. Some of these basic shades can register six variations in colour as the temperature rises, *i.e.* violet blue, when cold turns bright green at 311°F., olive green at 446°, dark grey at 545°, light grey at 554°, light brown at 572° and buff at 644°. The paint is also useful in tests of the heat-transfer qualities of lubricants, it is stated.

Air dehumidifier

A new dehumidifier claimed to remove 3 gal. of water from the air in 10,000 cu. ft. of space in 24 hr. has been developed by the Admiral Corp., Chicago, Illinois. It is an all-metal unit which draws air over a number of refrigerated coils; these condense the moisture in the air into a removable container. The dried air is then recirculated. The unit is said to be specially suitable for conditions of extreme humidity.

New zirconium process

Zirconium is claimed to be obtainable with a high degree of purity by a new process just patented. Zirconium is obtained, as in some other processes, by the reaction of one of its compounds with calcium, but the reaction takes place in a chamber filled with an inert gas and using h.f. induction heat. The patent number is 2,537,068 and Westinghouse Electric Corp. has obtained the rights.

Metal treatment patent

Over 12 years ago Richard F. Harvey, a metallurgist, developed a new hardening treatment which was first called 'step-quenching' and later became known as 'Martempering.' A U.S. patent application was made in 1940, but was eventually disallowed.

Recently two Congressional bills were filed to assist Mr. Harvey in obtaining a patent on this process.

This case is particularly interesting, because the last legislation of this kind, and probably the only other instance, dates back to 1878 when a patent was granted for a fire extinguisher originally applied for 41 years earlier.

Continuous manufacture of tetraethyl lead

Scheduled to come into operation next January, a plant for the production of 50,000,000 lb. p.a. of tetraethyl lead by a newly developed continuous process is to be constructed in New Jersey for the Du Pont Co.

Tetraethyl lead is at present made by a batch process.

CANADA

Glycol plant planned

A new plant to extract ethylene from petroleum refinery gases and produce ethylene glycol and ethylene oxide is to be erected in Montreal for the Dominion Tar & Chemical Co. Ltd., by E. B. Badger & Sons Co., through its associated Canadian company, Stone & Webster (Canada) Ltd. One of the principal raw materials required for the glycol plant is chlorine, which the company will obtain from its chlorine and caustic soda plant at Beauharnois, Quebec.

Export controls tightened

The Canadian Trade Department recently imposed export controls on 16 more commodities, bringing the total to more than 130. Export permits will be required for cobalt, tungsten and iron and steel fencing for shipments overseas and to the U.S. Lined steel tanks, antibiotics including penicillin, sulphur and white mineral oil will require permits for export to all countries except the U.S.

Opposition to aluminium project

American industrialists are trying to block the establishment of a $500,000,000 aluminium industry in British Columbia, according to the *Vancouver Sun*, and are doing everything within their power to prevent the Aluminium Co. of Canada Ltd. from setting up a plant there. Despite current shortages of aluminium, the U.S. fear future competition for as yet unbuilt plants, states the newspaper.

Chemicals-from-methane plant

The Celanese Corporation of America will shortly begin construction of a large petro-chemical and cellulose acetate plant in the Edmonton area of Alberta, Canada, which will materially accelerate the defence programmes of both the United States and Canada. A new Celanese affiliate, the Canadian Chemical Co. Ltd., has been formed in Alberta to build and operate the plant on a site already selected. Tapping the natural gas resources of the new and rapidly expanding petroleum fields in Alberta, the new company will manufacture many basic organic chemicals never before produced in Canada which will have broad applications in defence, war- and peace-time economies, and will contribute to the industrial self-sufficiency of the country. The company will use the Celanese process of direct oxidation of petroleum hydrocarbons. Acetate acid produced in the new plant will be combined with wood-pulp from the Columbia Cellulose Co. Ltd. plant at Prince Rupert, B.C., to manufacture cellulose acetate, the primary raw material for acetate yarns and plastics. Columbia Cellulose is another affiliate of the Celanese Corporation.

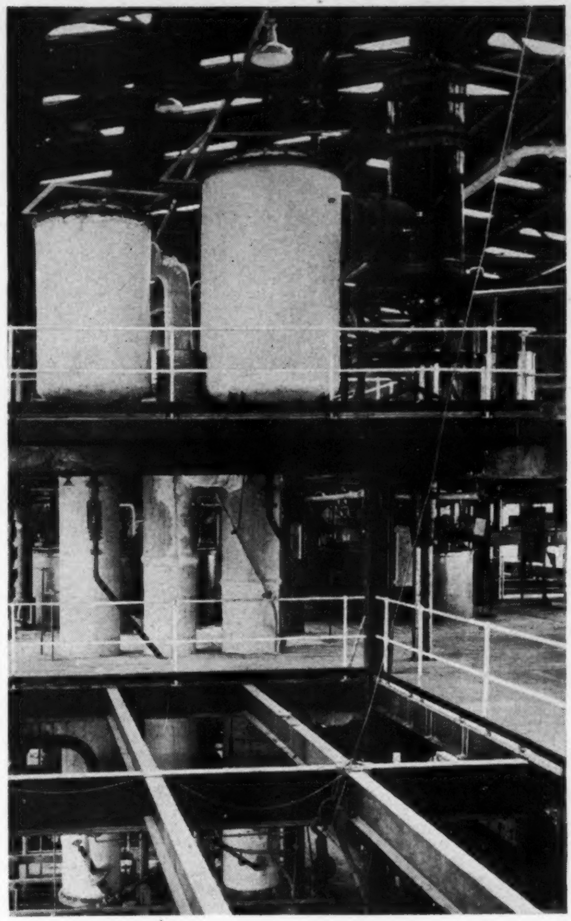
New paper mill for Labrador

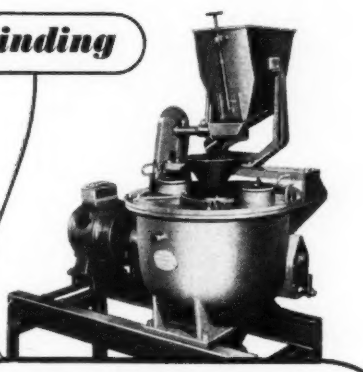
Dr. Arthur Seigheim, a German business man from Brussels, has discussed the erection of a new paper mill in Labrador with officials of the Department of Economic Development at St. John's, Newfoundland. Production at the existing paper mills at Corner Brook and Grand Falls is to be increased this year by a total of 80,000 tons. A new plant is being installed at the mills, owned by Bowaters and the Anglo-Newfoundland Development Co., respectively, to handle the bigger output.

MEXICO

New pulp factory

The investment of 100,000,000 pesos in a pulp factory in northern Mexico will be shared equally between Nacional Financiera, a bond and investment corporation in which the Government holds a controlling interest, and a group of private business men. The plant will be built at Temosochic, in the state of Chihuahua, a region whose forest reserves are sufficient to guarantee continuous production.





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